

June 1, 2001

Ms. Sandy Olinger (AMSAM-EN) Building 3206 Redstone Arsenal Huntsville, Alabama 35898

Sampling and Analysis Plan Determination of PCB TSCA Waste Quantities Building 3, St. Louis Army Ammunition Plant Contract No. DACW41-00-D-0019

Dear Ms. Olinger:

Arrowhead Contracting, Inc. (Arrowhead) is pleased to submit the enclosed Sampling and Analysis Plan (SAP) (Revision 1) for the Determination of PCB TSCA Waste Quantities at Building 3, Saint Louis Army Ammunition Plant, Saint Louis, Missouri. A distribution list for this document is attached.

If you should have any questions regarding the SAP, please call us at (913) 814-9994.

Sincerely,

Greg Wallace, R.G.

Project Manager

Enclosures:

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SAMPLING AND ANALYSIS PLAN DETERMINATION OF PCB TSCA WASTE QUANTITIES BUILDING 3 ST. LOUIS ARMY AMMUNITION PLANT ST. LOUIS, MISSOURI (Revision 1)

PRE-PLACED REMEDIAL ACTION CONTRACT CONTRACT NO. DACW41-00-D0019 TASK ORDER NO. 0002

Submitted to:

Department of the Army U.S. Army Engineer District, Kansas City Corps of Engineers 700 Federal Building 601 East 12th Street Kansas City, Missouri 64106

Department of the Army Aviation and Missile Command Building 3206 Redstone Arsenal Huntsville, Alabama 35898

Submitted by:



Arrowhead Contracting, Inc. 12920 Metcalf Avenue, Suite 150 Overland Park, Kansas 66213

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Sampling and Analysis Plan

This document constitutes a Sampling and Analysis Plan (SAP) for a field investigation that will support an engineering estimation of the quantity of selected polychlorinated biphenyl (PCB) contaminated material in Building 3 at the Saint Louis Army Ammunition Plant (SLAAP) located at 4800 Goodfellow Boulevard in Saint Louis, Missouri. These activities are being conducted in support of the proposed remedial action for Building 3.

The proposed remedial action at Building 3 includes the removal of PCB contamination in concrete, soil, and waste that is present at concentrations that are at or above 50 parts per million (ppm). The data and information that will be generated from the investigation outlined in this SAP will support the development of a Remedial Action Work Plan (RAWP).

This document was prepared on behalf of the U. S. Army Corps of Engineers (USACE), Kansas City District (CENWK) and the U.S. Army Aviation and Missile Command (AMCOM), Huntsville, Alabama under the Arrowhead Contracting, Incorporated (ACI) Pre-Placed Remedial Action Contract (PRAC) number DACW41-00-D0019, Task Order 0002. This SAP consists of two parts:

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Part I – Field Sampling Plan (FSP)
Part II – Quality Assurance Project Plan (QAPP)
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The FSP provides background information regarding previous operations at SLAAP and prior investigations, the data quality objectives for the proposed investigation, and a description of the procedures and protocols to be followed during the implementation of the proposed investigation activities. The QAPP provides the quality assurance/quality control guidelines for the collection and analysis of all environmental samples.

This document has been prepared in accordance with the provisions of USACE Engineering Manual (EM) 200-1-3.

Part I - Field Sampling Plan

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- Inspection Checklist Composite Concrete Floor Sampling
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- Nonconformance Report
- Field Work Variance
- Daily Quality Control Report
- Corrective Action Request

List of Acronyms_

ACI Arrowhead Contracting, Inc.

AMCOM Aviation Missile Command

ATCOM Army Aviation and Troop Command

ATSDR Agency for Toxic Substances and Disease Registry

AVSCOM Aviation Systems Command

CENWK U.S. Army Corps of Engineers, Kansas City District

CENWO U.S. Army Corps of Engineers, Omaha District

CAR corrective action request

COC chain-of-custody

CQAB U.S. Army Corps of Engineers, Chemistry Quality Assurance Branch of

the Waterway Experiments Station Environmental Laboratory

DQCR Daily Quality Control Reports

DFW definable feature of work

DoD U.S. Department of Defense

DQO data quality objective

EBS Environmental Baseline Study

EM Engineering Manual

EPA U.S. Environmental Protection Agency

FWV Field Work Variance FSP Field Sampling Plan

 H_0 null hypothesis ID inner diameter

IDW investigation derived waste

kg kilogram

mg/kg milligram(s) per kilogram
μg/l microgram(s) per liter
mg/l milligram(s) per liter

mm millimeter

NCR Nonconformance Report
NON Notice-of-Noncompliance

OSHA Occupation Safety and Health Association

PCB polychlorinated biphenyl

PPE personal protective equipment

PRAC Pre-Placed Remedial Action Contract

List of Acronyms (continued)

QAPP Quality Assurance Project Plan

QC quality control

QCP Quality Control Plan

QMP Quality Management Plan
RAWP Remedial Action Work Plan

RCRA Resource Conservation and Recovery Act

SAP Sampling and Analysis Plan

SHERP Safety, Health, and Emergency Response Plan

SLAAP St. Louis Army Ammunition Plant

SLOP St. Louis Ordnance Plant

sq. ft. square feet

SVOCs semi-volatile organic compounds

TCLP Toxicity Characteristic Leaching Potential

TSCA Toxic Substances Control Act
USACE U.S. Army Corps of Engineers

1.0 Introduction

The purpose of this Field Sampling Plan (FSP) is to establish the sampling strategy, sample locations, and the procedures and protocols to be followed during a sampling effort in support of an engineering estimation of the quantity of selected polychlorinated biphenyl (PCB) contamination in Building 3 at the Saint Louis Army Ammunition Plant (SLAAP), Saint Louis, Missouri (refer to Figure 1-1). The scope of the sampling activities were developed based on findings and recommendations included in the following documents:

- Final Environmental Baseline Survey Report, Saint Louis Army Ammunition Plant, St. Louis, Missouri (Tetra Tech, 2000)
- Alternatives Evaluation for Removal of PCBs, Saint Louis Army Ammunition Plant, St. Louis, Missouri (Arrowhead, 2001)

This document has been organized into eleven sections. The contents of each section are discussed below.

- Section 1.0 Introduction
 - Presents an introduction to site history, physical features of the building, current understanding nature and extent of PCB contamination, risk-based cleanup goals, and regulatory drivers.
- Section 2.0 Project Organization and Responsibilities
 - Identifies organizations, roles, and responsibilities for key personnel to be used during the field activities.
- Section 3.0 Sampling Program Rationale
 - Presents a sampling strategy based on the data quality objective (DQO) process.
- Section 4.0 Field Activities
 - This section presents a description of the field activities, the rationale for conducting the activities, the field protocols to be used during the activities, and laboratory analysis for the planned field sampling activities.
- Section 5.0 Sample Chain-of-Custody/Documentation
 - Presents details regarding sample documentation including field logbooks, sample labels, sample collection field sheets and chain-of-custody.
- Section 6.0 Sample Packaging, Shipping, and Archiving
 - Presents details regarding sample packaging, shipping and archiving.

- Section 7.0 Investigation Derived Wastes
 - Presents details regarding handling, storage, and disposal of investigation derived waste.
- Section 8.0 Contractor Quality Control
 - Presents details regarding contractor quality control.
- Section 9.0 Field Corrective Actions
 - Presents a discussion of corrective actions for any non-conformances identified in the field.
- Section 10.0 Project Schedule
 - Presents a schedule for the field and activities and reporting associated with this SAP.
- Section 11.0 References
 - Presents references that are relevant to the basis of this FSP.

1.1 Site History

In 1941, the St. Louis Ordnance Plant (SLOP) was constructed on a 276-acre parcel of property near what is now the intersection of Goodfellow Boulevard and Interstate I-70. SLOP was constructed to produce 0.30- and 0.50-caliber munitions in support of World War II. In 1944, approximately 21 acres in the northeast portion of SLOP was converted from small arms munitions production to 105-millimeter (mm) Howitzer shell production and was designated as SLAAP. Currently, the SLAAP property consists of eight unoccupied buildings that were used to house SLAAP main operating processes. This study focuses solely on Building 3, also historically referred to as Building 202ABC. The processes completed in Building 3 included shell-shaping, heat-treating, cleaning, painting, and packaging shells for shipment. Following World War II, SLAAP was placed on standby status, only to be reactivated to support the Korean Conflict (from November 1951 through December 1954) and the Vietnam War (from November 1966 through December 1969).

In 1984, Building 3 was renovated to include office space for personnel from the U.S. Army Aviation Systems Command (AVSCOM). The building was occupied in this capacity until 1996. In 1989, the Department of Defense (DoD) determined that SLAAP was no longer needed for munitions support and all industrial equipment was removed from the facilities. Since 1998, Building 3 has been vacant and under the control of AMCOM.

1.2 Physical Features of the Building

Building characteristics, historical uses, historical processes, and hazardous material information for Building 3 are summarized in Table 1-1. Building 3 consists of a basement, first floor, second floor, and 2 penthouses (third floor). Figures 1-2 and 1-3 show the location where process operations took place on the first and second floors, respectively, of the building. The majority of the flooring in the building is concrete; however, much of the basement flooring is dirt. As described in sections to follow, much of the original flooring on the first and second floors is covered with a concrete cap that was placed following a removal of a portion of the original concrete floor surface. The primary features in the basement area include a chip chute area and two catch basins.

1.3 Current Understanding of Nature/Extent of Environmental Contamination

Oils containing PCBs were used in Building 3 primarily as a coolant in the milling, lathing, and smoothing processes associated with munitions production. PCBs were first discovered in Building 3 in creosote-treated wood flooring blocks during renovation activities in March 1991. The U.S. Environmental Protection Agency (EPA) Region VII was notified of the discovery and, in turn, issued a notice of noncompliance (NON) under the Toxic Substances Control Act (TSCA) in May 1991 (TSCA Docket Number VII-91-304).

The NON stated that the facility was not in compliance with the National Spill Clean-Up Policy for PCBs (40 C.F.R. Part 761.125) and requested documentation of the following four items:

- Evidence of the removal and proper disposal of all contaminated mastic and wood from both floors of Building 3.
- Evidence of the removal and proper disposal of all contaminated plastic and fiberboard from the file storage area.
- Decontamination of all non-porous surfaces to less than 10 micrograms per 100 square centimeters (μg/100 cm²) and verification of the same by submitting results of analyses from post decontamination wipe sampling to this office (EPA Region 7).
- Decontamination of all porous surfaces (concrete) to less than 10 parts per million (ppm) PCBs as determined by destructive sampling (core sampling). Please submit a statistically based sampling plan to this office prior to such sampling and coordinate sampling activity with this office so an inspector can be on-site to witness the activity and obtain split samples for EPA analysis.

Since the NON has been issued, a number of decontamination and confirmatory sampling activities were conducted on the first and second floors to assess the nature an extent of the residual PCB contamination. For example, Rust Remedial Services, Inc. (Rust), formerly Chemical Waste Management, Inc., performed decontamination and confirmatory sampling activities for the PCB contamination in Building 3 from September 1991 through August 1994. Decontamination activities included removal of the PCB-contaminated wood blocks, scarification of the concrete floor surfaces, and washing of block walls on the first and second floors of the building. Additional decontamination activities were performed in the summer of 1996 to remove PCB contamination from the first floor. The removal of PCB contamination from the flooring was performed by scarification methods. The location of the scarified areas is unknown. A 2- to 4-inch thick concrete cap was placed over most of the first and second floors after the confirmation sampling was completed. However, it should be noted that the concrete cap was not placed in the area of the office space on the second floor and the areas of the where the transformers were located.

As part of the remedial approach for Building 3, a health-based risk assessment was completed to determine risk-based cleanup levels for the basement and the first and second floors of Building 3. The risk assessment concluded that residual contamination in the building did not present an unacceptable health impact and that further remediation was not necessary. The Agency for Toxic Substances and Disease Registry (ATSDR) did not endorse the health-based risk assessment. Samples collected from porous (concrete) surfaces and the non-porous (steel) surfaces in support of the risk assessment evaluation indicated residual PCB contamination was still present at concentrations that exceeded federal guidelines.

On August 7, 1997, the U.S. Army Aviation and Troop Command (ATCOM) sent a letter to EPA Region VII documenting its agreement to complete the following tasks for Building 3:

- Paint the walls and ceilings and cap the floor with concrete,
- Isolate the chip chute by constructing a wall in the basement,
- Develop a sampling plan and perform a health risk assessment to be reviewed by the appropriate Army agency,
- Take ambient air samples to measure PCB levels after completion, and
- Meet with EPA Region VII to determine if any future action is needed.

To date, the NON issued by the EPA is still unresolved. EPA has indicated in more recent discussions that the clean-up standards set forth in the 1998 TSCA Amendments (see Section 1.4) supercede the standards as set forth in the NON.

More details regarding each of the decontamination and confirmatory sampling events are provided in the *Final Environmental Baseline Survey Report for the St. Louis Army Ammunition Plant, St. Louis, Missouri, December 28, 2000* (Tetra Tech, 2000). Finally, in August 1997, ATCOM directed painting of the walls and ceilings and capping of the floors with concrete to prevent exposure to the residual PCB contamination.

Analytical data have been used from the aforementioned decontamination and sampling episodes in an attempt to quantify the levels of residual PCB contamination on each of the floors within Building 3. In general, nature and extent of PCB contamination remaining in Building 3 can be summarized as follows:

- The majority of the PCB contamination within Building 3 is associated with the concrete flooring on the first and second floors (see Figures 1-2 and 1-3). Concentrations range from 2 ppm (detection limit) to 730 ppm (in the area of the former chip chute). The degree to which the PCBs penetrated into the concrete flooring is unknown. Presumably, the depth of PCB penetration is greatest in processing areas where the oils accumulated beneath the oil soaked wood blocks. Many of the remaining areas are likely contaminated at relatively shallow depths, such as the walkways, canteens, and restrooms. Concentrations in these areas are likely attributed to foot traffic from SLAAP personnel.
- The chip chute area where the PCB contamination is present in the walls, flooring, and in a pile of waste material (most likely residual cuttings/shavings from the operation). It is unknown whether the PCB contamination extends into the soil underlying the waste pile.
- Selected columns in the basement where the PCBs seeped downward from the first floor. There are no data available to characterize the extent and magnitude of this contamination.
- Selected areas of the basement flooring where the PCBs appeared to have leaked through cracks, piping, drains, or along joints in the first floor. Oil staining on both the concrete and dirt flooring has been observed in the areas beneath the former process areas on the first floor. There are also two former catch basins along the north wall of the basement that contain piles of stained soil that may represent waste from the operation. There are no data available to characterize the extent and magnitude of potential PCB contamination, if present, in these areas.

- The location of four transformer vaults in the basement. There are no data available to characterize the extent and magnitude of this contamination.
- Spill areas in the penthouse where PCBs may have leaked from motors. It is assumed that this contamination is confined to a relatively small area and that the depth of contamination is relatively shallow. There are no data available to characterize the extent and magnitude of this contamination.
- A soil sample collected from a soil boring (3SB-3) located immediately outside Building 3 and adjacent to the loading dock at the Chip Chute detected PCBs at a concentration of 830 mg/kg.

Various levels of other contaminants such as asbestos and pesticides have been detected within Building 3 (Tetra Tech, 2000). These other contaminants, however, are not applicable to the PCB remedial action and, therefore, are not addressed as part of this study. These contaminants will be addressed as part of a proposed demolition of the building.

1.4 Risk-Based Cleanup Goals

CENWK has prepared a risk analysis for potential future exposures to PCBs in the building. The analysis was based on the most prevalent PCB contaminant within Building 3 (Aroclor 1248) and established acceptable risk at the 10⁻⁶ level. Three different receptor scenarios were evaluated including:

- A future industrial worker who works in the building and comes into contact with PCBs on floor and wall surfaces (comparable to the TSCA high occupancy scenario),
- A future industrial worker who spends part of his/her time working in and around the uncovered contaminated soil in the basement of the building (comparable to the TSCA low occupancy scenario), and
- A future demolition worker who is exposed to contaminated concrete debris during demolition of the building at some future date (this scenario is believed to be the most conservative of the scenarios evaluated because it results in significant direct contact with the contaminant).

The risk-based cleanup goals established for each of the aforementioned scenarios are as follows:

- $15 \mu g/100 cm^2$ on concrete surfaces for the future industrial worker,
- 26 ppm in basement soil for the future industrial worker, and
- 16 ppm in concrete for a worker involved in the demolition of Building 3.

EPA Headquarters has not approved these risk assessment values.

1.5 Regulatory Drivers

PCB contamination within Building 3 is subject to the rules and regulations set forth in TSCA, as amended by the "Mega Rule" in 1998. These regulations provide standards governing the distribution of PCB-contaminated items, including acceptable cleanup approaches and standards, disposal requirements, and sampling and analysis protocols.

Section 761.20 of TSCA prohibits the "distribution in commerce of PCBs at concentrations of 50 ppm or greater". Because PCB contamination exists within Building 3 at concentrations that exceed the 50 ppm threshold criterion, the sale of the property is prohibited until those concentrations are reduced to levels deemed acceptable by EPA Headquarters.

Because PCB contamination is present at concentrations that exceed 50 ppm, all portions of the resulting contamination must be remediated to an acceptable level. Remedial activities may be self-implemented in accordance with regulations set forth in §761.61(a) of TSCA. Under these regulations, cleanup standards are established for porous surfaces (including concrete) as follows:

- High Occupancy Areas: 1 ppm without restriction, 10 ppm with a 10-inch cap, and a surface cleanup standard at $10 \mu g/100 cm^2$.
- Low Occupancy Areas: 25 ppm, or 25-50 ppm if fenced and marked, 25-100 ppm with a 10-inch cap, and a surface cleanup standard at $100 \, \mu g/100 cm^2$.

It is important to point out that these cleanup standards are applied with regard to all contaminated material. In other words, cleanup of all PCB contamination exceeding the cleanup standards is required, not just those portions that exceed the 50 ppm triggering criterion. An alternate (risk-based) cleanup number that is higher than the aforementioned cleanup standards may be established if deemed by EPA as sufficiently protective (see Section 1.4 for risk-based numbers).

The definition of high and low occupancy areas is provided in §761.3 of the regulations. Consistent with this definition, the first, second, and penthouse floors of Building 3 are considered high occupancy areas while the basement of Building 3 is considered a low occupancy area. The high and low occupancy criterion does not necessarily apply to the transfer of the property.

Contamination that is removed from the site must be handled in accordance with the Bulk PCB Remediation Waste Criteria. Material containing PCBs at 50 ppm or greater must be disposed in a TSCA-permitted facility (or in a landfill at a similar Resource Conservation and Recovery Act (RCRA) facility). Material containing PCBs at concentrations less than 50 ppm may be placed in a municipal or non-industrial, non-hazardous landfill that is permitted to accept low concentrations of PCBs. In addition, Subpart O of the regulation provides a leaching test option to allow the use of solid waste landfills for disposal of PCB-containing waste that are not readily leaching to the environment, i.e., concentrations in leachate less than 10 micrograms per liter (μ g/l).

2.0 Project Organization and Responsibilities

Table 2-1 identifies organizations, roles, and responsibilities for key personnel to be used during the Building 3 characterization project. Off-site analytical services will be provided by a USACE-approved laboratory (to be determined). Quality Assurance (QA) split samples will be analyzed by the USACE laboratory located in Omaha, Nebraska.

3.0 Sampling Program Rationale

The sampling strategy described in this FSP is based on the Data Quality Objective (DQO) process presented in *EPA Soil Screening Guidance: Technical Background Document* (EPA, May 1996). Based on this guidance, a sampling strategy has been developed and organized consistent with the steps of the DQO process:

- State the problem
- Identify the decision
- Identify inputs to the decision
- Define the study boundaries
- Develop a decision rule
- Specify limits on decision errors
- Optimize the design for obtaining data

Each of these steps is discussed below.

3.1 Data Quality Objectives Process

3.1.1 State the Problem

The objectives of the sampling program are to collect sufficient data to:

- Define the area and volume of PCB contamination at concentrations of 50 ppm or greater that may be present in concrete, soil, and waste material at Building 3.
- Determine the chemical composition of the Chip Chute waste pile and the soils in the catch basins for evaluating disposal options.
- Provide information for determining the appropriate method for disposing waste building materials during a planned remedial action at Building 3.
- Provide information for assessing the health and safety issues associated with disturbance of building materials (i.e. dust) during a planned remedial action at Building 3.
- Provide information to verify that oil staining is a reliable indicator for identifying TSCA waste in basement soils.

The last four objectives are considered incidental to the first objective since it is the PCB contamination that is driving the remediation of the building. Hence, the remaining portions of this section will address decisions regarding the PCB contamination.

3.1.2 Identify the Decision

The decision to remediate or not remediate concrete, soil, and waste material in Building 3 as part of the proposed remedial action will be based on whether PCB concentrations in these materials are at 50 ppm (the threshold value for waste that would be disposed of at a TSCA facility) or greater. If so, the materials will be addressed as part of the remedial action. The decision regarding the proper method of disposing building materials and other waste materials (i.e. Chip Chute waste pile and catch basin material) is contingent upon the PCB concentration and concentration of RCRA constituents (metals and semi-volatiles). Decisions related to health and safety requirements are based on the chemical contaminants in the building materials that could potentially expose workers during remedial activities.

3.1.3 Identify Inputs to the Decision

This step in the DQO process requires identifying the inputs to the decision process, including the basis for investigation and the applicable field sampling and analytical methods. The inputs for deciding whether to investigate are based on visual observations made during recent site visits and on information contained in the following documents:

- Existing characterization of the nature and extent of PCB contamination in Building 3 as defined in the Final Environmental Baseline Survey Report, Saint Louis Army Ammunition Plant, Saint Louis, Missouri (Tetra Tech, December 2000)
- Alternatives Evaluation for Removal of PCBs, Saint Louis Army Ammunition Plant, Saint Louis, Missouri (Arrowhead, March 2001)

For sampling of the selected areas of Building 3, the inputs for deciding whether to investigate include the following:

- The recommended remedy in the Alternatives Evaluation includes removal and disposal of PCB contamination at or in excess of 50 ppm from Building 3. Material containing PCBs at 50 ppm or greater must be disposed in a TSCA-permitted facility (or in a landfill at a similar RCRA facility). Material containing PCBs at concentrations less than 50 ppm may be placed in a municipal or non-industrial, non-hazardous landfill that is permitted to accept low concentrations of PCBs.
- Figures 1-1 and 1-2 depict the most current interpretation of the nature and extent of PCB contamination in the concrete flooring, first and second floor, respectively. The identified areas of contamination in these figures were developed by AMCOM based on statistical sampling data collected by others prior to placement of the concrete cap. These data represent average concentrations within selected areas of the building. Each area was

divided into as many as 48 sectors for purposes of the confirmation-sampling program. Three sample aliquots (designated A, B, and C) were collected from each sector. All of the sample aliquots labeled "A" from each sector within an area were composited together and analyzed for PCBs. The aliquots designated "B" and "C" were composited and analyzed in the same fashion. Given this approach, it stands to reason that a measured concentration of 50 ppm within an area would indicate that at least one of the sectors in the area contains PCB contamination in excess of 50 ppm. However, there is less certainty regarding the remaining areas of the building where lower levels of PCBs were measured. For example, it is possible that one or even more of the sectors within these areas could contain PCB in excess of 50 ppm, even though the concentration of the composite sample is less than 50 ppm.

- EBS sampling data, which indicates the concentrations in the concrete (flooring, columns, and walls) and waste material in the chip chute area, exceed 50 ppm.
- Second floor office areas that have not been sampled to date will be included in the investigation since they correspond to former process areas.
- General statements in the EBS noting visual oil staining on the concrete columns as well
 as wipe samples of the column surfaces in the basement, which indicate the presence of
 PCBs. Visual oil staining has been observed on columns in the basement. The columns
 on the first and second floor are painted and therefore it is not possible to see if any
 staining is present.
- The reported former presence of four transformer vaults in the basement. There are no data available to characterize the nature and extent of potential PCB contamination in these areas. It is assumed that the transformer may have used PCB-containing oil.
- The report and observation of possible oil leaks from the formers motors located in the penthouse. There are no data available to characterize the nature and extent of this potential contamination.
- Observations of oil stained soils within catch basins in the basement.
- A small area of soil contamination located adjacent to Building 3 (near the chip chute area) that was identified in the EBS.
- During recent site visits, there were a number of small areas in the basement where oil staining was observed on concrete flooring, soil flooring, and concrete columns.
- There were a few cracks and small areas in the first and second floors where oil staining was observed during recent site visits.
- The assumed depth of contamination in the concrete floors is based on knowledge of the location of different types of work areas (areas where process activities were conducted,

areas where SLAAP's process personnel traveled about the building, and waste storage areas; i.e. the chip chute area). It is suspected that the depth of PCB contamination at concentration in excess of 50 ppm is greatest in the areas where the PCB contamination may have pooled for extended periods of time (i.e. areas where the process equipment was stationed) and in areas where the waste material was stock piled (i.e. the chip chute). It is suspected that PCB contamination at concentrations in excess of 50 ppm in areas exposed to foot traffic, in walls, and in minor spill areas (i.e. areas where transformer and motors were staged) may be relatively shallow (i.e. less than 1 inch). It should be noted that a concrete cap was placed over the original flooring on the second and first floors. The cap thickness appears to vary from approximately 2 to 4 inches based on visual observation.

3.1.4 Define the Study Boundaries

This step in the DQO process defines the sample media of interest (areas and depths of concern), subdivides areas of concern into manageable units, and specifies temporal or practical constraints on the data collection.

3.1.4.1 Media of Interest

The media of interest includes those materials that contain PCB contamination at concentrations that are equal to or greater than 50 ppm. Based on the discussion above, it is anticipated that the PCB concentration will vary with depth dependent on the type of area and the orientation of the surfaces (i.e. process areas, traffic areas, walls, columns). The depth of interest will also be dependent on the practical limits of the available remediation techniques and safety concerns associated with these techniques. The following criteria are applicable to the selection of sampling depths:

- It is anticipated that the practical depths limits of a partial floor removal is approximately 4 to 6 inches for the first floor (total floor thickness is approximately 16 inches) and approximately 2 inches below the original floor surface for the second floor (total floor thickness is approximately 8 inches). It is suspected that rebar is present in the second floor concrete starting at about 2 inches below the original surface. It is likely that the rebar in the first floor is much deeper. It should be noted that a 2- to 4-inch concrete cap is present throughout both floors (excluding the office areas on the second floor).
- It is assumed that PCB contamination at concentrations that is 50 ppm or greater in traffic areas is present in only the uppermost inch of flooring below the concrete cap.
- It is assumed that the depth of PCB contamination at concentrations that are 50 ppm or greater in the process areas can be greater than one inch below the original floor surface.

- In the basement, the depth of PCB contamination at concentrations that are 50 ppm or greater in the columns and the miscellaneous spill areas (in the former areas of the transformers and motors and small oil stained concrete areas) is assumed to be 1 inch or less.
- Based on visual observations of oil staining, concrete columns in the basement appear to contain contamination over their entire length.
- Soils adjacent to the former chip chute area will be sampled from 0 to 6 inches and 12 to 18 inches in depth. The sampling methodology and density are described in Section 4.3.5.
- Catch basins will be sampled at depths of 0 to 1 feet and 1 to 2 feet below grade.
- The flooring material in the chip chute area is unknown at present, and may be comprised of concrete (unknown thickness) or soil. The depth of PCB contamination at concentrations that are at 50 ppm or greater is unknown.
- Soils in the basement that contain visual observation of oil staining, and areas below oil staining on the basement ceiling will be sampled. Soil samples from these areas will be collected from 0 to 6 inches and 6 to 12 inches in depth.

Based on the criteria listed above, Table 3-1 presents the intervals in the selected areas of concern that will be sampled to define the vertical extent of PCB contamination:

3.1.4.2 Areas of Concern

Table 3-2 identifies the Areas of Concern that will be investigated. The limits of the Areas of Concerns on the first and second floors were developed based on the information shown in Figures 1-2 and 1-3, respectively, and correspond to areas where PCBs were detected at concentrations greater than 5 ppm during the EBS. The locations of all other Areas of Concern were identified based on visual observations made on recent site visits and from information in the EBS. The locations of the Areas of Concern (proposed sampling locations) are shown on Figures 3-1, 3-2, and 3-3 for the basement, first floor, and second floor, respectively.

The flooring to be investigated on the first and second floor will be divided into 20 ft. by 20 ft. grid sectors based on the locations of building columns. Note that the limits of sampling the former process areas have be expanded to also include grid sectors located adjacent to the sectors designated as containing PCB contamination at concentrations above 5 ppm on Figures 1-2 and 1-3. In addition, it is assumed that there are 20 areas on the first and second floors containing

miscellaneous PCB oil stains (located outside the process or traffic areas already designated for sampling) that will be investigated. Also, the second floor office space has not been evaluated for the presence PCBs; these areas will be investigated as part of this study using the same grid sector spacing as the remainder of the second floor. It is also assumed that the miscellaneous oil-stained areas on the first and second floor will be investigated in the same fashion as the traffic or process areas on the first and second floor (i.e. contamination profiles are believed to be similar). Furthermore, it is assumed that there are 30 areas in the concrete floor portion of the basement and 30 areas in the soil floor portion of the basement containing miscellaneous PCB oil stains that will be investigated. Miscellaneous oil-stained areas are assumed to be relatively limited in areal extent (i.e. 100 ft²). Non-stained soil in the basement will be sampled to verify that oil staining is a reliable indicator of TSCA contaminated areas. Four samples will be collected from non-oil stained areas

The sampling of the columns in the basement, Chip Chute waste pile, Chip Chute flooring, Chip Chute walls, former transformer locations in the basement, and miscellaneous oil stained areas in the basement motor area and catch basins will be conducted on a discrete basis. The areas of suspected PCB soil contamination located outside the building will be sampled based on a grid system.

3.1.4.3 Constraints on Data Collection

The sampling will be confined to areas where PCB contamination is suspected of being present at concentrations of 50 ppm or greater. This target concentration has been selected to support remediation of materials that will be disposed at a TSCA facility.

3.1.5 Develop a Decision Rule

Based on EPA guidance, the following decision rule has been adopted for this FSP:

If the mean contaminant concentration of total PCBs exceeds the action level (as defined in Section 3.1.6) in a selected area (sector) or at a discrete sample location, then the materials associated with that sector or location will be subject to disposal at a TSCA facility.

The following criteria were used for purposes of defining sectors on concrete flooring:

- The concrete flooring on the first and second floors has been divided in 20 ft. by 20 ft. sectors defined at the corners by the existing columns.
- The area of concrete flooring beneath the former transformer and motor locations may be reduced or enlarged to include areas where visual oil staining is observed.
- The concrete flooring in the basement will be selected for sampling based on visual observation of oil staining.

The following criteria were used to define all other areas of concern:

- The area and volume of soil to be remediated that is located outside the building will be defined based on samples located in a sampling grid.
- Columns in the basement will be sampled at locations where visual oil staining is observed. Columns in the selected process areas will be sampled near the column base.
- The Chip Chute Area will be divided in five sectors; the northwest wall, the southeast wall, the northeast (back) wall, waste pile, and floor beneath the waste pile.
- The short concrete walls that surround each area and the pile of stained soil with each basin define the two catch basins.

3.1.6 Evaluate Decision Errors and Optimize the Design

The PCB sampling data will be used to support a decision about whether an area will be remediated. Because of variability in contaminant concentrations within an area, practical constraints on sample sizes, and sampling or measurement error, the data collected may be inaccurate or non-representative and may mislead the decision makers into making an incorrect decision. A decision error occurs when sampling data mislead decision makers into choosing a course of action that is different from or less desirable than the course of action that would have been chosen with perfect information.

The EPA guidance, *Verification of PCB Spill Cleanup by Sampling and Analysis* (EPA, 1985), recognizes that data obtained from sampling and analysis are never perfectly representative and accurate, and that the costs of trying to achieve near-perfect results can outweigh the benefits. Consequently, uncertainty in data must be tolerated to some degree. The DQO process controls the degree to which uncertainty in data affects the outcomes of decisions that are based on those data. This step of the DQO process allows the decision maker to set limits on the probabilities of making an incorrect decision.

The DQO process utilizes hypothesis tests to control decision errors. When performing a hypothesis test, a presumed or baseline condition, referred to as the "null hypothesis (H_0) ", is established. This baseline condition is presumed to be true unless the data conclusively

demonstrate otherwise, which is called "rejecting the null hypothesis" in favor of an alternative hypothesis.

When the hypothesis test is performed, two possible decision errors may occur:

- 1. Decide not to remediate an area (i.e., "walk away") when the correct decision (with complete and perfect information) would be to "remediate"
- 2. Decide to remediate when the correct decision would be to "walk away."

The first error would be a <u>false negative</u>, i.e., failure to detect the presence of PCB levels above the allowable limit. The second error would result in a <u>false positive</u>, i.e., concluding that PCBs are present at levels above the allowable limit when, in fact, they are not.

To minimize the likelihood of false negatives, the areas will be subdivided into sectors no larger than 400 ft². To protect against false positive findings due to analytical error, the measured PCB level in a single sample must exceed some value greater than 50 ppm for a finding of contamination. Assuming a 0.5% false positive rate and standard statistical techniques, the action level for a single sample would be:

$$(0.8)(50) + (2.576)(0.2)(0.8)(50) = 60 ppm$$

where 0.8 (80%) represents the accuracy of the analytical method, 50 ppm is the allowable limit for a single sample, 2.576 is a coefficient from the standard normal distribution, and 0.2 (20%) is the standard deviation of the analytical method. Thus, if the measured level in a single sample is 60 ppm or greater, one can be 99.5% sure that the true level is 50 ppm or greater. However, in order to provide an even greater level certainty against false negatives, the action level will be set at 50 ppm.

To economize on the number of samples and analyses while providing areal coverage of the sampling area, the sectors of the concrete flooring will be subdivided into four quadrants. A sample aliquot will be collected at the center of each quadrant. The four aliquots will be composited and analyzed as one sample. If the sample result is 50 ppm or greater, then the sector will be designated for remediation. This concentration will represent an average of the four aliquots.

Letting X ppm be the measured concentration in the composite sample, then if $X \le (50/4) = 12.5$ ppm, then individual samples are statistically predicted to be less than 50 ppm. If 12.5 ppm < X < 50 ppm, no conclusion is possible based on the analysis of the composite and the four aliquots must be analyzed individually to reach a decision.

All other sample locations (the Chip Chute area, catch basins, the columns, the soil located outside the building, and the miscellaneous oil-stained areas in the basement) will be selected on a discretionary basis to define the nature and extent of the PCB contamination.

3.2 Sample Collection Summary

This section introduces the basic methodology for PCB sampling. The specific procedures associated with these methods are discussed in Section 4.3. Table 3-4 presents a summary of the PCB sampling methods for each area of concern.

Composite samples of the concrete flooring for PCBs will be collected using coring methods to the depths specified in Table 3-3. A core sample will be collected at each aliquot location. The sample collection procedure is discussed in Section 4.3.1. The former process and traffic areas on the first and second floors (see Figures 3-2 and 3-3) will be sampled according to the following protocol:

- Every other sector within the overall sampling area will be sampled initially, such that each unsampled sector is adjacent to a minimum of two sampled sectors (i.e. checkerboard pattern) (Refer to Figures 3-2 and 3-3).
- To determine the sampling outcome of unsampled sectors, the following criteria will be used:
 - A sector will be deemed contaminated (with no further sampling) if sampled sectors on opposite and adjacent sides are found to be contaminated.
 Contaminated areas are defined as containing PCBs above the action level defined in Section 3.1.6.
 - If the sampled sectors on opposite and adjacent sides have different outcomes (i.e. one contaminated, the other uncontaminated), then the outcome of the unsampled

sector is deemed inconclusive. Accordingly, this sector will be sampled to determine the appropriate outcome.

• If the corner sector of the sampling area is the unsampled sector, then the outcome is considered conclusive only if both adjacent sectors have the same outcomes.

Discrete samples of concrete flooring from miscellaneous oil-stained areas on the first and second floor will also be collected using coring methods. However, Discrete concrete floor samples in the basement, former transformer areas (basement), and former motor area (penthouse) will be collected using drilling methods to the depths specified in Table 3-3. The drilling procedure is discussed in Section 4.3.2.

Sample of material from the Chip Chute waste pile and within the basement catch basins will be collected with a stainless-steel bucket auger. The samples from the Chip Chute waste pile will be comprised of representative material from a sampling interval of 0 to 2 feet. Samples from the catch basins will be comprised of material from the 0 to 1 feet and 2 to 3 feet depth intervals. The sample collection procedure for the waste sampling is discussed in Section 4.3.3.

If present, the concrete beneath waste pile will be sampled by drilling methods (refer to Section 4.3.2). The core samples will be collected at two locations (0-1 inch) corresponding to the locations where the waste pile samples will be collected. Whether a concrete floor is or is not present beneath the waste pile, a sample of the soil beneath the waste pile will be collected at the same locations. Soil samples will also be collected from a grid area located outside the building (adjacent to the Chip Chute Area), from miscellaneous oil-stained areas identified on the soil portion of the basement floor, and from non-oil-stained areas of the basement floor. Two intervals (0 to 6 inches and 12 to 18 inches) will be sampled at each location. Soil sampling procedures are discussed in Section 4.3.4.

The walls in the Chip Chute area will be sampled using drilling methods. Two samples will be collected from each of the three walls. A sample will be collected near the center of each half of the wall. Columns in the basement will also be sampled using drilling methods. Wall and column samples will be collected from the depth interval 0-1-inch. The sample collection procedure for concrete walls and columns is discussed in Section 4.3.2.

In addition to PCB sampling, samples of concrete, soil, and waste material will be collected for waste characterization and health and safety characterization. Tables 3-5 and 3-6 present a summary of the sampling approach, including estimated sampling quantities for waste characterization, and health and safety characterization, respectively. Specific procedures for waste characterization and health and safety sampling are discussed in Section 4.0.

4.0 Field Sampling Activities

This section presents a description of the field sampling activities and protocols to be implemented during the sampling effort at Building 3. The activities discussed in this section include building surveying, sample layout, PCB sampling, waste pre-determination sampling, health and safety pre-assessment sampling, and equipment decontamination.

It should be noted that a project-specific Safety, Health and Emergency Response Plan (SHERP) has been prepared for Building 3 field activities. Topics presented in the SHERP including, among other things, potential hazards associated with field activities at Building 3, recommended hazard control measures, and requirements for personal protective equipment (PPE). Personnel involved with the field activities described in this FSP shall comply with provisions in the SHERP. Due to the expected generation concrete dust during field activities, Level C PPE (including half-face respirators with HEPA filters) is anticipated for certain aspects of the field work. However, PPE requirements may be adjusted based on actual field conditions and results of monitoring activities (refer to the SHERP).

4.1 Building Contamination Pre-Inspection

At the start of the field activities, field personnel will perform a pre-inspection of Building 3 for the presence of oil staining on columns or floors. The locations, dimensions, and description of the stained areas will be documented and sketched. The locations of the stained areas will then be marked for subsequent sampling. The building pre-inspection information will be used to identify biased locations for sampling of the floors and columns as described in Sections 4.3.2 and 4.3.3.

4.2 Layout of Sampling Locations

Following the building pre-inspection, field personnel will identify all sampling locations (with the exception of the locations below the waste pile). Each location will be marked with the corresponding sample ID (refer to Section 5.3) using chalk, spray paint, or crayon. A member of the sampling team will review the locations to ensure that they do not conflict with building utilities. Additionally, sampling personnel will check the initial locations of samples outside the building relative to underground utilities. Underground utilities will be located using available building maps or by contacting Missouri One Call. If conflicts with utilities are identified, the sample location(s) will be moved to the nearest safe location. Field personnel will then ground-truth the locations of all samples by measuring from existing features (columns, walls, ceilings,

doorways, etc.). The sample locations will be recorded on a base map of each floor of the building.

4.3 PCB Sampling

Concrete, waste, or soil samples will be collected from the areas shown on Figures 3-1, 3-2 and 3-3. These figures exclude the miscellaneous oil-stained areas to be identified during the building pre-inspection and additional concrete floor samples that may be collected based on the results of initial floor samples. All samples will be submitted to a USACE-approved laboratory as discussed in the QAPP. The overall sampling program involves the collection of five types of samples:

- Composite Concrete Samples (Floors)
- Discrete Concrete Samples (Floors)
- Discrete Concrete Samples (Walls and Columns)
- Waste Samples
- Soil Samples

The methods used to collect each of these sample types are described below. In addition, Table 3-4 presents a summary of each area of Building 3 to be sampled and the proposed sampling method.

The total number of samples, analytical parameters, and analytical methods associated with each sample type is presented in Table 4-1 of the QAPP. The quantity and type of QA/QC samples associated with each type of sample is given in Table 6-1 of the QAPP. Sample containers, preservation procedures, holding times, and sample volumes associated with all of the sample types are given in Tables 5-1, 5-2, and 5-3 of the QAPP.

4.3.1 Concrete Sampling Using Coring Methods

Composite samples will be collected from floors on the first and second floors of the building. The composite samples will be collected form two types of areas: former process areas and former traffic areas (refer to Figures 3-2 and 3-3 and Tables 4-1 and 4-2). As described in Section 5.3, a grid system will be established that subdivides the floors into sectors using building columns as reference points. A single composite sample from each sector/location will be collected that consists of sample aliquots from four quadrants within the sector/location. The center point of each quadrant will constitute the location of the aliquot. Composite concrete floor samples will be collected as follows:

- At the aliquot location, the concrete floor will be cored to the appropriate depth (2 below the interface of the concrete cap and original floor in the former traffic areas or 3 inches below the interface of the concrete cap and original floor in the former process areas) using a 1 to 2 inch inner diameter (ID) core sampler.
- The concrete core sample will then be saw-cut into individual sections corresponding to the sample depth interval (0–1 inches and 1-2 inches in the former traffic areas; 0-1 inches and 2-3 inches in the former process areas).
- The individual core section will then be drilled or crushed using appropriate mechanical equipment (i.e. pulverizer, drill press, crushing unit). The powdered material (pulverized cuttings/particles) generated during the drilling/crushing operation will be collected in a bowl or other container.
- Five (5.0; plus or minus 0.05) grams of the powdered material will be weighed on a laboratory-grade scale.
- The 5.0-gram sample will then be placed into the sample container designated for the specific location/sector and depth interval. The remaining powder and unused core section will be placed into a secondary container and labeled with the quadrant ID (refer to Section 5.3). This material will be retained for possible future analysis.
- The remaining aliquots from the same location/sector and depth interval will be prepared as described above. Five (5.0; plus or minus 0.05) grams of powdered material from each of four (4) aliquots will be placed into the sample container.
- The sample, consisting of four (4) aliquots, will then be composited by aggressively shaking the material within the sample container. A sample container of adequate volume (4-ounce glass jar) will be used to ensure that there is sufficient space within the container for shaking and thoroughly mixing the material.
- After compositing, the sample container will be labeled with the sample ID for the specific location/sector and depth interval (refer to Section 5.3).
- The sampling equipment (core sampler, saw blade, drill bit, crushing equipment, sample collection bowls, etc.) will be decontaminated between each sample location, aliquot, and depth interval to prevent cross-contamination of samples. Equipment decontamination procedures are described in Section 4.6.

Discrete concrete samples will be collected from miscellaneous oil-stained areas on the first and second floor. These samples will be collected using the same procedure described above, except that the sample will not consist of multiple aliquots. A single sample will be cored to depth and

cut into sections corresponding to the sampling intervals (0 to 1 inch and 1 to 2 inch below the concrete cap). The individual sections will be drilled or crushed and the pulverized material (approximately 30 grams) collected and placed into the sample container. The sample will be labeled to identify the location and depth interval in accordance with Section 5.3.

4.3.2 Concrete Sampling Using Drilling Methods

Discrete concrete samples will be collected from floors in the basement, transformer vaults, and penthouse (former motor area) at oil-stained locations identified during the building preinspection (refer to Section 4.1). Discrete samples will also be collected from building columns in the basement based on oil staining identified during the building pre-inspection. In the Chip Chute area, samples will be collected from each of the three walls. Two samples will be collected from each wall – one sample from the center of each half of the wall. If the chip chute walls are found to be composed of brick and mortar instead of concrete the samples will be collected in the same manner as samples from concrete walls.

These concrete floor, column, and wall samples will be collected as follows:

- Using a drill, holes will be drilled in the vicinity of the sampling location to the specified depth.
- A sufficient number of holes will be drilled (to a depth of approximately one-inch) to produce the required sample mass (approximately 30 grams)
- For floor samples, upon completion of the 0-1 inch interval, the drilled hole will be cleaned out (by brushing, blowing with compressed air, or other appropriate methods) prior to initiating the sample from the 1-2 inch interval.
- The powdered material generated during the drilling process will be collected and placed directly into the sample container designated for the specific location.
- After collection, the sample container will be labeled with the sample ID for the specific location (refer to Section 5.3).
- The sampling equipment (drill bit, etc.) will be decontaminated between each sample location to prevent cross-contamination of samples. Equipment decontamination procedures are described in Section 4.6.

4.3.3 Waste Sampling

Waste samples will be collected from two locations at the Chip Chute waste pile (one interval: 0 - 2 feet) and two locations at each of the catch basins (two intervals: 0 - 1 feet and 2 - 3 feet):

- Samples will be collected using a stainless-steel bucket auger.
- Waste material from the selected interval at each location will be thoroughly mixed in a stainless-steel bowl with a stainless-steel spoon.
- After mixing, a sufficient quantity (enough to fill each sample container) of waste material will be placed in the sample container designated for the specific location. The container will be labeled with the sample ID corresponding to the location (refer to Section 5.3).
- Extra material will be returned to the waste pile. The waste pile will eventually be removed and disposed, pending waste characterization analysis.
- The sampling equipment (bucket auger, mixing bowl, etc.) will be decontaminated between the two sampling locations as described in Section 4.6.

4.3.4 Soil Sampling

Soil samples will be collected at twelve (12) sample locations within an area outside Building 3, adjacent to the Chip Chute. These samples will be distributed on 10-foot grid spacing within an area of concern with dimensions of 30 feet by 20 feet. Soil samples will also be collected below the Chip Chute waste pile. If concrete flooring is present below the waste pile, soil samples will be collected via the holes created from concrete coring. If concrete is not present below the waste pile, samples will be collected from the soil approximately below the location of prior waste pile samples (refer to Section 4.3.4). Furthermore, soil samples will be collected from the basement floor at both oil-stained areas (identified during the building contamination pre-inspection) and non-oil-stained areas. Soil samples will be collected as follows:

- Discrete soil samples will be collected using a stainless steel hand auger and/or a small barrel drive sampler. The auger or driver will be advanced to the appropriate depth interval (0-6 in. and 12-18 in.).
- Soil from each depth interval will be thoroughly mixed in a stainless steel bowl with a stainless steel spoon.

- After mixing, a sufficient quantity of soil (enough to fill the sample container) will be placed in a sample container designated for the specific location and depth interval. The container will be labeled with the sample ID corresponding to the location and depth interval (refer to Section 5.3).
- Extra soil from the sample boring will be returned to the borehole.
- The sampling equipment (auger, mixing bowl, etc.) will decontaminated between each sampling location and between each depth interval as described in Section 4.6.

4.4 Remediation Waste Pre-Determination Sampling

Based on the results of PCB sampling, areas of Building 3 containing PCBs at concentrations at or exceeding the action level will subject to remediation. The remediation will involve the removal and disposal of select portions of Building 3 concrete, waste (Chip Chute and catch basins) and soil. To select the appropriate disposal facility and/or to meet the waste disposal acceptance criteria of the disposal facility, the chemical composition of the remediation-derived waste materials (concrete, waste pile, soil) must be determined. Waste materials must be properly classified in accordance with RCRA and TSCA (i.e. hazardous waste, special waste, TSCA waste, non-hazardous waste). The collection and analysis of PCB characterization samples, as discussed in Section 4.3, will be sufficient for assessing disposal options related to TSCA. However, additional samples will need be collected and analyzed to satisfy RCRA requirements. The receiving disposal facility will likely require that representative samples be collected immediately prior to shipment. For example, if remediation waste materials are being shipped by rail car, a representative sample will need to be collected from each rail car. As a preliminary assessment (pre-determination) of the waste characteristics of potential remediation wastes to be disposed off-site, samples of concrete, soil, and waste from Building 3 will be collected and analyzed during this investigation. The sampling data will be useful for future remediation disposal planning, as it will provide a general indication of whether remediation materials are likely to be classified as RCRA hazardous waste. The selection of a proper disposal facility will be contingent upon the TSCA and RCRA profiles of the waste materials. Samples collected for remediation waste pre-determination will be analyzed for semi-volatile organic compounds (SVOCs) and metals per the Toxicity Characteristic Leaching Procedure (TCLP) (refer to QAPP). Samples will also be analyzed for petroleum hydrocarbons, as that information may be required for future waste profiling. Table 3-5 summarizes the sampling approach and estimated quantities for remediation waste characterization samples. Sampling methods will be consistent with Section 4.3. It is anticipated that remediation pre-determination samples will be collected at the same time as the corresponding PCB samples.

4.5 Health and Safety Pre-Assessment Sampling

The remediation and possible future demolition of Building 3 will generate significant quantities of dust and debris. During remediation and demolition activities, workers could potentially be exposed to these materials and associated contaminants. Therefore, as part of this investigation, additional samples will be collected and analyzed to characterize the contaminants that may be present in dust and debris. Contaminant concentrations will be compared to OSHA permissible exposure limits. The collection and analysis of PCB characterization samples, as discussed in Section 4.3, will be sufficient for assessing the health and safety concerns associated with exposure to PCBs. However, samples will need to be collected to assess other contaminants that could present a health and safety concern. Potentially hazardous constituents associated with Building 3 materials are anticipated to be SVOCs and metals. Table 3-6 summarizes the sampling approach and estimated quantities for health and safety pre-assessment sampling. Sampling methods will be consistent with Section 4.3. It is anticipated that health and safety pre-assessment samples will be collected at the same time as the corresponding PCB samples.

4.6 Silica Monitoring

Levels of silica dust in ambient air will be monitored as part of the SHERP. Details of silica monitoring are discussed in the site-specific SHERP.

4.7 Equipment Decontamination

Field personnel will exercise caution in decontaminating coring equipment, drill press, crushing equipment, concrete saw, mixing bowls, mixing spoons, and hand tools. Sampling equipment will be decontaminated between each sample location, aliquot, and depth interval (soil samples). The decontamination procedure will include a wash with Alconox soap and water followed by a rinse with the analytical grade methanol and then with deionized/distilled water. All decontamination fluids will be containerized and managed as investigation-derived waste (IDW) as discussed in Section 7.0. At the conclusion of the project, samples of decontamination water will be collected and analyzed to assess the disposal options.

5.0 Sample Chain-of-Custody and Documentation

During sampling activities, the samples must be traced from the time the samples are collected until laboratory data are issued and samples appropriately disposed. Initial information concerning collection of the samples will be recorded in a field logbook. Information regarding the transfer, handling, and shipping of all samples will be recorded on a Chain-of-Custody (COC) included in Appendix A.

The sample custodian will be responsible for initiating and filling out the COC. The sampling team members are responsible for the care and custody of the samples collected until the samples are transferred to another individual or shipped to the analytical laboratory. The sampling team, under the direction of the Field Supervisor, is responsible for enforcing COC procedures during fieldwork. The sample custodian when samples are relinquished to anyone else will sign the COC, with date and time. The COC will accompany the samples at all times. All individuals who subsequently take possession of the samples will also sign, with date and time, the COC.

Each cooler containing samples sent to the analytical laboratory will be accompanied by the COC. Laboratory personnel are responsible for the receipt and entry of samples into the laboratory that have been submitted under a COC document. Additionally, samples received will be entered into the laboratory COC system by properly documenting and maintaining COC from the moment that they take custody of the sample until the sample is properly disposed.

5.1 Field Logbook

Field logbooks will be maintained to record all pertinent information. Entries will be as descriptive and detailed as possible so that a particular situation can be reconstructed without reliance on the collector's memory. Field logbooks (which will consist of a 5 x 7 1/2-inch bound book with consecutively numbered pages) will be kept by a field representative.

The cover of each field logbook will contain the following information:

- Project name and number
- Book number
- Activity type
- Start date

• Stop date.

Entries to a field logbook will be made daily and, at a minimum, will consist of the following:

- Date
- Start time
- Weather
- All field personnel present
- Visitors to the site (time, name, and company)
- Level of personnel protection used
- Type of activity conducted
- Sampling location
- Sample identification number
- Description of sampling point
- Method of sampling
- Type of sample
- Air monitoring readings, if applicable
- Pertinent field observations
- Field measurements, if applicable
- Description of all related activities
- Signature of the person making the entry.

All measurements made and samples collected will be recorded. All entries will be made in indelible ink. No erasing of entries will be permitted. If an incorrect entry is made, the data shall be crossed out with a single strike mark and initialed. Entries will be organized into easily understandable tables, if possible.

At each location where a sample is collected or a measurement made, a detailed entry of the sampling location, equipment used to collect the sample(s), depth interval, time of sample collection, number of samples, types of analysis, and number and types of sample containers will be recorded. All equipment used to make measurements, if necessary, will be identified, including the date on which the equipment was calibrated.

Field documentation requirements associated with site health and safety is presented in the SHERP.

5.2 Photographs

Color digital photographs will be taken prior to, during, and after conducting field activities. Photographs will be tracked with a numbered photograph log that will include the project name, date, and description of activity or location (e.g., oil stained area, waste pile, core sampling, and soil sampling).

5.3 Sample Numbering System

The sample numbering system will provide a tracking number to allow retrieval of the sample and exact identification of the sample location. Each type of sample collected will be identified by a two-letter prefix code as follows:

- CF Concrete Floor Sample
- CW Concrete Wall Sample
- CC Concrete Column Sample
- SS Soil Sample
- WP- Waste Pile Sample
- IW Investigation Waste Sample

A numeric designation will follow the sample type prefix to identify which floor the sample was collected from. The numbers 0, 1, and 2 will be used to denote the basement, first floor, and second floor, respectively. An alphanumeric designation will follow the numeric floor designation to identify the sample location of sample collected on the first and second floor. The alphanumeric designation will correspond to the column identification (i.e. "C11" would correspond to the column located in row C, column 11; refer to Figures 1-2 and 1-3) located in the northern most corner of the sector sampled. The sample of the columns on the first and second floor will be identified by the column location (i.e "C11" would correspond to the column located in row C, column 11; refer to Figures 3-2 and 3-3). For example, CF1-D12 indicates a concrete floor sample from the first floor, in the sector located adjacent to column D12.

The aliquots from each sector will be designated with the letter A, B, C, or D depending on which quadrant it was collected in. For example, CF1-G14A indicates a concrete floor (first floor) sample from quadrant "A" of the sector located adjacent to column G14. The remaining quadrants (B, C, and D) will be designated in counter clockwise fashion from quadrant "A".

The sample identification number will include a two-digit number following the sample type and location prefixes to identify the sampling interval. For example, CF1-D12-23 indicates a concrete floor sample (first floor) from the sector located adjacent to column D12 at a sampling interval of 2 to 3 inches below the original floor surface.

For field duplicates, the sample identification number will have a "500" added to make it unrecognizable to the subcontract laboratory. For example, sample identification CF1-D12-23 would be CF1-D512-23 if it were a field duplicate. For sample splits to the USACE quality assurance laboratory, the sample identification will have a "D" added as a suffix. For example, CF1-D12-23 would be CF1-D12-23D if it were a split sample.

For rinsates, the sample identification number will have a "R" added as a suffix to the sample identification number of the sample collected prior to the rinsate. For example, CF1-D12-23R would be a rinsate collected immediately after the concrete floor sample CF1-D12-23.

5.4 Sample Documentation

Sample documentation will be conducted in accordance with the following subsections.

5.4.1 Sample Labels

Each sample collected for chemical analysis, or archived for possible future analysis, will be placed in the appropriate container(s) and labeled at the time of sample collection with the following information:

- Arrowhead project number and name
- Sample number
- Date and time of collection
- Required analyses and methods
- Matrix sampled
- Type of preservative, if applicable
- Volume of sample and container type
- The name of the sampler
- Initials of the sampler and date.

5.4.2 Sample Collection Field Sheets

An example of a Sample Collection Field Sheet to be used to document pertinent information associated with the various samples is presented Appendix A.

5.4.3 Chain of Custody Procedure

The COC procedures are as follows:

- At the time of sample collection, the COC is completed for the sample collected.
- The sample custodian will cross-check the form for possible errors. Corrections will be made to the record with a single strike mark and dated and initialed. All entries will be made in blue or black ink. The COC will be signed when the samples are relinquished.
- A shipping bill will be completed and the shipping bill number recorded on the COC prior to enclosing inside a clear plastic bag and attaching it to the inside of the cooler lid.

When transferring custody of the samples, the individual relinquishing custody of the samples will verify sample numbers and condition and will document the sample acquisition and transfer by signing, with date and time, the COC. The sample custodian will group samples for shipment to the analytical laboratory and complete a COC for each cooler of samples. Samples will be packaged for shipment and dispatched to the analytical laboratory with a COC accompanying each cooler.

Custody seals will be used to ensure that sample shipping containers have not been opened during shipment and prior to receipt at the off-site laboratory. The following information will be included on the custody seals:

- Signature of the sample custodian
- Date when the sample package is sealed.

All seals will be completed using indelible ink. The seals will be affixed to the front and back of the cooler, at the interface of the cooler and the lid. The placement of the seals will be in a manner that breaking the seals would be necessary in order to open the sample cooler.

In conjunction with data reporting, the analytical laboratory will return the original or a photocopy of the original COC to the Contractor for inclusion into the project file.

All samples collected will remain in the possession of the sample custodian until shipment. Secured areas will be used for interim storage if necessary. If coolers (used for sample storage) must be left unattended for extended periods of time, signed custody seals will be placed on the front and back of each cooler or the cooler will be stored under lock until shipped to the off-site laboratory.

When the analytical laboratory receives the sample coolers, a receipt for sample for will be initialed. The laboratory will document the sample condition upon receipt. All receipt nonconformance situations will be reported immediately to the Field Supervisor.

5.4.5 COC Documentation

A copy of each COC will be retained by the sample custodian for the project file, and the original sent with the samples. For sample packages sent by carrier to a laboratory off-site, shipping receipts will be retained as part of the documentation for the COC records.

6.0 Sample Packaging and Shipping

This section describes packaging and shipping procedures for collecting environmental samples. Samples will be shipped off-site according to applicable guidance documents and U.S. Department of Transportation (DOT) regulations. To minimize sample container breakage and provide adequate sample temperature during shipment, sample containers will be prepared and packaged according to the following procedures:

- Secure sample bottle lids (Note: Teflon cap liners will be used for all samples submitted for PCB analysis). Ensure that the sample label is securely attached by placing clear tape over the label.
- Place custody tape over the sample container lid or cap.
- Place sample bottles in Styrofoam sleeves (if provided); or place sample bottles in "Ziplock" clear plastic bags and wrap them with protective packing material.
- Tape the drain hole shut on the inside and outside of a waterproof metal (or equivalent strength plastic) cooler.
- Line the sides and floor of the cooler with protective packing material.
- Line the cooler with a large plastic bag.
- Place containers upright in the cooler in such a way that they do not touch.
- Packing material will be placed in appropriate locations to minimize potential container breakage during shipment. Care will be taken so that the packing material does not thermally insulate the containers from the ice placed in the shipping container.
- Pack the area surrounding the samples with ice (either chemical ice packs or ice cubes sealed in plastic bags).
- Fill the remaining space in the cooler with cushioning material.
- Close the large plastic bag in the cooler and tape or secure shut.
- Place the completed COC and other paperwork in a sealed, clear plastic bag and tape the bag to the inside lid of the cooler. (Note: The original COC will accompany the shipment, and copies will be retained by the sampler for return to project management and the project file).

- Wrap the cooler completely around with strapping tape at two locations. Do not cover any labels.
- Place the address label of the shipment destination on top of cooler.
- Affix signed custody seals on the cooler at the interface between the cooler and the lid, both in the front and the backside. Cover the seals with wide, clear tape.
- Make a copy of the shipping bill for the project file and place the original in a clear envelope secured to the outside of the cooler lid.

Samples will be sent to an off-site laboratory by use of an overnight courier delivery service. Prior to shipment of samples, arrangements will be made with the laboratory to receive and analyze the samples.

Laboratory specific receiving and handling procedures will be described in the Laboratory Quality Assurance Plans.

Key personnel contacts are provided below:

ANALYTICAL LABORATORY

TBD

CENWK

U.S. Army Corps of Engineers ATTN: CENWK-PM-ED (Dan Mroz) Technical Manager 700 Federal Building, 6th Floor 601 E. 12th Street Kansas City, MO 64106 (816) 983-3368

U.S. Army Corps of Engineers ATTN: CENWK-EC-DC (Francis Zigmund) Project Chemist 700 Federal Building, 6th Floor 601 E. 12th Street Kansas City, MO 64106 (816) 983-3905

CQAB

Ms. Laura Percifield 420 South 18th Street Omaha, NE 68102 U.S. Army Corps of Engineers ATTN: CENWK-EP-ED (Kurt Baer) Project Engineer 700 Federal Building, 6th Floor 601 E. 12th Street Kansas City, MO 64106 (816) 983-3392 (402) 444-4314

ARROWHEAD

Greg Wallace, Project Manager/Field Supervisor 12920 Metcalf, Suite 150 Overland Park, Kansas 66213 (913) 814-9994 (913) 461-3828 (cell phone)

Doug Ronk, Sample Custodian 12920 Metcalf, Suite 150 Overland Park, Kansas 66213 (913) 814-9994 (913) 461-3805 (cell phone) Scott Siegwald, SHSO and QC Inspector 12920 Metcalf, Suite 150 Overland Park, Kansas 66213 (913) 814-9994 (913) 461-3804 (cell phone)

7.0 Investigation-Derived Waste Management

IDW generated during project activities will include decontamination (rinse) water, soil from soil borings, concrete from concrete sampling, and PPE. General procedures for managing IDW are as follows:

- Decontamination fluids and fluids generated during sampling activities will be containerized in a holding tank or in 55-gallon drums. Containerized decontamination fluids will be labeled and inventoried. Labels shall, at a minimum, define the contents, the date the IDW was collected, and the reason for containerization. An up-to-date container inventory will be maintained on site that documents the type of container, the contents of the container, date of arrival at storage area, and the container status (e.g., awaiting analytical results). In addition, routine visual inspections of the storage area will be made to identify areas of potential leaks or spills. At the conclusion of the field sampling activities, samples of the containerized fluids will be submitted to the analytical laboratory for analysis of PCBs, Total SVOCs, and Total Metals as discussed below.
- Unused portions of soil samples will be returned to the sampling location (i.e. placed back into the bore hole).
- Unused portions of concrete from concrete samples and miscellaneous concrete cuttings will be placed back into the core holes from which they were collected. This material will be disposed during remediation of Building 3 in a manner consistent with remediation waste materials. This determination will be based on waste characterization sampling as described in Section 4.4.
- Personnel protective clothing will be placed in plastic trash bags and disposed as municipal waste.

Waste minimization will involve the following objectives:

- Segregate clean fill from contaminated soil and water. Clean fill is defined as containing PCBs at levels below the cleanup standard of 1ppm.
- Minimize volume by cleaning, compacting, drying, and decanting
- Separate soil waste media from water waste
- Plan not to mix contaminant in containers; segregate wastes by contaminants
- Clean contaminated PPE if possible and dispose as solid municipal waste

- Use waste minimization as a design criteria and for planning for design life cycles, per U.S. Department of Defense (DoD) directives
- When possible, budget final waste disposal costs within each activity budget and each activity schedule to avoid accumulating waste.

Decontamination fluids will be sampled via an access port at the top of the drum or holding tank using a decontaminated bottle sampler. The sample will be transferred to the appropriate sample containers (refer to Section 5.0 of the QAPP). A Sample Collection Field Sheet (refer to Appendix A) will be completed and the following information recorded in a field logbook:

- Date/time of sampling
- Sampling team personnel
- Sample number
- Quantity of decontamination fluid in container
- Location of container sampled
- Other data as required.

Samples of decontamination fluids will be packaged and shipped to the designated analytical laboratory as discussed in Section 6.0 this FSP. Final disposition of the drummed wastewaters will be determined based on the results of the laboratory analysis.

8.0 Contractor Quality Control

This section provides the criteria for the performance of inspections of each Definable Feature of Work (DFW) associated with the field activities. Inspections are the processes whereby the Quality Control (QC) Inspector, by examination or measurement, determines that an activity complies with the specified quality requirements. The inspection system is based on the USACE three-phase system of control to cover the activities. The three-phase inspection system consists of preparatory, initial, and follow-up inspections for applicable DFWs.

8.1 Definable Features of Work

A DFW is defined as a major work element that must be performed in order to execute and complete the project. It consists of an activity or task that is separate and distinct from other activities and requires separate control activities. The following DFWs have been identified for the planned field activities:

- Layout and Building Survey
- Concrete Floor Sampling
- Concrete Wall and Column Sampling
- Waste Pile Sampling
- Soil Sampling
- Investigation Derived Waste Sampling

A detailed inspection checklist for each of these DFWs is included in Appendix A.

8.2 QC Inspections

The QC Inspector will coordinate inspection activities with the Project Manager/Field Supervisor, subcontractors, and field personnel. Inspection activities will be performed on a periodic basis.

8.2.1 Preparatory Inspections

Preparatory inspections will be performed prior to the initiation of all DFWs. The preparatory inspection is performed in advance of any work being performed to enable all involved parties to determine whether or not everything is properly in place and ready to initiate the work activity. This inspection will be conducted by the QC Inspector and will be attended by field personnel and subcontractors. The preparatory inspection will be scheduled prior to the start of the DFW.

All affected parties will be notified in advance of the inspection to coordinate their participation. The preparatory inspection will include, but is not limited to:

- Review of pertinent contract requirements and plans
- Review of required control inspections and test requirements
- Review of reports, forms, and checklists that need to be filled out during the activity
- Review of subcontracts and purchase orders
- Review of required licenses, permits, and utility notifications
- Establish that required planning documents have been reviewed and approved by USACE and regulators
- Establish that the required materials and equipment for commencement of the DFW are on-hand or available and are in accordance with plans and calibration requirements
- Establish that the preliminary work required to begin the DFW is complete and conforms to approved plans
- Schedule the date that the initial inspection, if required, will be performed
- Review and discuss the SHERP requirements for the DFW.

For analytical activities, the QC Inspector will contact the laboratory to insure they are ready to begin accepting samples and to review any questions regarding the requirements of the QAPP or the subcontract.

8.2.2 Initial Inspections

Initial inspections will be conducted at the initiation of a DFW. The initial inspection will provide the opportunity for the QC Inspector to observe the actual initiation of the work activity and the individual segments of the DFW. The inspection will be performed on a representative sample of work to evaluate the following criteria:

- Compliance with the plans and other contract requirements
- Acceptable levels of workmanship

- Identify use of defective or damaged materials
- Identify improper procedures or methods
- Acceptable test or inspection results
- Compliance with the SHERP.

8.2.3 Daily QC Inspections

Daily QC inspections of field activities will be performed on a daily basis when work on a DFW is in progress. The Daily QC inspections will be performed until all work on a DFW is completed. The following items will be performed during the Daily QC inspection:

- Verify compliance with the plans and other contract requirements
- Verify level of workmanship, if applicable
- Verify test or inspection results
- Verify nonconformance issues are identified, corrected, and re-inspected
- Verify compliance with the SHERP.

8.2.4 Documentation

The preparatory, initial, and follow-up inspections will be documented on forms. Example Preparatory, Initial and Daily QC Inspection Checklist are provided in Appendix A. The Daily QC Inspection Checklist will be attached to the Daily Quality Control Report (DQCR) (refer to Section 8.3) and submitted to the USACE on a weekly basis during performance of the activity. If a final inspection for either a specific task or the entire project is required, this information will be provided on the Final Inspection Form presented Appendix A.

If the inspection process identifies a nonconforming condition, it will be documented, tracked, and corrected. Non-conformance Reports (NCR) and Corrective Action Requests (CARs) will also be attached to the Daily Quality Control Report (refer to Section 9.0).

8.3 Daily Quality Control Reports

DQCRs will be prepared to document field activities performed. Quality control personnel will prepare DQCRs with input from the Field Supervisor, sampling personnel, and others conducting

the field activities. The DQCRs will contain the following information pertaining to the field sampling activities:

- Weather information at the time of sampling
- Sample collection field sheets
- Copies of field logbooks
- Copies of COC forms
- Field instrument calibration forms
- Field instrument measurements
- Verbal instructions received from CENWK or AMCOM personnel
- Problems encountered during sampling
- Field Work Variances
- Forms included in this SAP.

Attachments to the DQCR will include:

- Daily QC Inspection Checklist
- CAR, if necessary
- NCR, if necessary
- Daily Chemical Data Report (refer to Section 15.0 of the QAPP)

9.0 Field Corrective Actions

Corrective actions will be implemented by the Contractor or its subcontractors to correct nonconformance issues identified during QC inspections or during the course of conducting activities. A nonconformance is defined as a deficiency in implementation of a procedure or standard that renders the quality of an item or activity unacceptable or indeterminate with respect to the acceptability criteria. Correction of nonconformance issues will be focused at determining the cause of the deficiency and instituting actions to correct the deficiency and prevent recurrence.

Corrective actions will be implemented and documented via a CAR. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are deemed insufficient, work may be stopped through a stop-work order issued by the Contractor Project Manager and/or the CENWK Project Manager

9.1 Nonconformance Reporting

Noncompliance with specified criteria will be documented through a formal nonconformance control and corrective action program. Personnel who identify a nonconformance are responsible for notifying the Contractor Project Manger of the nonconformance. The Contractor Project Manager will discuss the nonconformance with USACE on-site representative to determine if the nonconformance has been properly described and that applicable project requirements or criteria have not been met to warrant issuance of a NCR (refer to Appendix A). The Contractor Project Manager will immediately notify the CENWK PM of any major or critical deficiencies (i.e., deficiencies requiring re-sampling, re-analysis of samples, or re-drilling/coring) identified during the course of project execution.

9.2 Nonconformance Disposition and Tracking

Corrective actions required to bring nonconforming conditions into compliance will be approved by the Contractor Project Manager prior to implementation. Corrective actions will be documented in a field CAR, which will be attached to DQCR. NCRs will remain on open status and tracked until the corrective actions have been implemented and verified acceptable by the Contractor Project Manager. If appropriate, the Contractor Project Manager will ensure that no additional work associated with the nonconforming activity is performed until the corrective actions are completed. This will be implemented through a stop-work order issued by the Contractor Project Manager.

9.3 Field Work Variances

Changes to approved plans or procedures may be required when events occur or presumed information must be altered based on actual conditions encountered during the course of field activities. Request for approval to vary from approved plans, specifications or procedures will be submitted to the CENWK with a Field Work Variance (FWV) (refer to Appendix A). Minor variances can be implemented in the field prior to receipt of written approval of the FWV when approved by the USACE on-site representative. Minor variances are defined as those variances that do not affect project cost, schedule, quality or quantities. Major variances require written approval prior to implementation. Major variances impact cost, schedule, quality, and quantities and vary from the approved plans, specifications, or procedures. FWVs will be submitted to the USACE COR for approval. All changes as a result of FWVs will be documented in a final report.

10.0 Project Schedule

The procurement of Subcontractors, equipment, and supplies will begin approximately 4 weeks prior to fieldwork. Fieldwork is tentatively scheduled to begin on June 17, 2001 (refer to Figure 10-1). The estimated time to completion for fieldwork is 6 weeks. The final results of chemical analysis will be completed approximately 2 weeks after the field is completed. It is anticipated that the results of this investigation will be summarized in a Data Report. A draft version of the Data Report will be completed approximately one month after the all the chemical analysis results are received.

Task/Deliverable	Scheduled Date of Completion/Date of Submittal				
Final Field Sampling Plan	June 1, 2001				
Safety Health and Emergency Response Plan (SHERP)	10 days prior to the scheduled field activities				
Field Sampling Activities	June 17 through July 18, 2001				
Final Laboratory Results Received	14 days following end of field sampling activities				
Draft Data Report	45 days after receipt of final laboratory results				
Final Data Report	30 days after receipt of draft report comments				
Monthly Progress Reports	Second full work week of each month				

11.0 References

Arrowhead Contracting, Inc. (Arrowhead). 2001. <u>Alternatives Evaluation for Removal of PCBs</u>, Saint Louis Army Ammunition Plant, St. Louis, Missouri. December.

Tetra Tech EM, Inc. (Tetra Tech). 2000. <u>Final Environmental Baseline Survey Report</u>, Saint Louis Army Ammunition Plant, St. Louis, Missouri. December.

United States Environmental Protection Agency (EPA). 1985. <u>Verification of PCB Spill</u> <u>Cleanup by Sampling and Analysis</u>. August

United States Environmental Protection Agency (EPA). 1996. <u>Soil Screening Guidance:</u> <u>Technical Background Document</u>. May.

Tables

Table 1-1
Summary of Physical Features of Building 3

Building Characteristics					
	Basement: 37,000 square feet (ft ²)				
Araa	First Floor: 168,000 ft ²				
Area	Second Floor: 154,780 ft ²				
	Penthouse: 6,813 ft ²				
Style	Two stories, basement, and two penthouses (third floor)				
	Steel frame and roof beams on reinforced concrete piers and spread				
Construction Materials	footings; masonry walls; and a prefabricated concrete roof. The eastside				
	addition has the same structure, but also is covered with asbestos siding.				
Construction Date	Built in 1941, retooled (including eastside addition) in 1944. Renovated				
Construction Date	to create office space in 1984 and 1985.				

Historical Use					
	1941 to 1944: SLOP (0.30-caliber munitions production)				
Occupants/Lessees	1944 to 1984: SLAAP (105-millimeter (mm) Howitzer shell production				
Occupants/Lessees	– intermittent production)				
	1985 to 1996: SLAAP (AVSCOM office space)				
	1941 to 1944: 0.30-caliber munitions production				
	1944 to 1945: 105-mm Howitzer shell production				
Operational Periods	1952 to 1954: 105-mm Howitzer shell production				
	1966 to 1969: 105-mm Howitzer shell production				
	1985 to 1996: Office space				
	Historical Processes				
	Processes completed in Building 3 consisted of shell shaping, heat				
	tracing, cleaning, painting, and packaging for shipment. Metal chips				
	and fragments produced as a result of the shell machining processes				
Process Description	were collected on the first and second floors and disposed in the chip				
	chute. The chip chute is an open chute along the north wall that opened				
	to the basement in Building 3. From the basement, the metal chips were				
	transferred to a railcar via conveyor for off-site disposal.				
	Process machinery included lathes, drill presses, milling machines,				
	grinders, heat-treating furnaces, wash racks, welders, shapers, shot-				
Process Machinery	blasting equipment, paint spray booths, transformers, air compressors,				
	and auxiliary equipment (dust collection devices, elevators, and				
	conveyors).				
Process Utilities	Process utilities included water, steam, compressed air, soluble oil,				
	quench oil, paint, natural gas, telephone service, and electricity.				
Hazardous Material Information					
Possible Hazardous	Cutting (soluble) oil*, quench oil (No. 6 fuel oil), hydraulic oil, solvents				
Material Used	(toluene), asbestos, lead-based paint, and pesticides.				

^{*} contained polychlorinated biphenyls (PCBs)

Table 2-1
Summary of Project Organization and Responsibilities

KEY PERSONNEL	ORGANIZATION	ROLE	RESPONSIBILITIES
Sandy Olinger Dan Mroz	AMCOM CENWK	Project Manager Project Manager	 Contract management Technical oversight Right of entries Request LIMS number from CQAB
Laura Percifield	CQAB	Laboratory Supervisor	QA sample analysisAssign LIMS number for off- site analysis
Greg Wallace, R.G.	Arrowhead	Project Manager/Field Supervisor	 Primary contact point with CENWK and AMCOM Overall responsibility for all phases of work Oversight of filed activities Technical direction to field subcontractors and field personnel Layout of sample locations and building survey
Scott Siegwald	Arrowhead	Site Health and Safety Officer/Quality Control Inspector	 Directing overall chemical QA\QC program Oversight of Off-Site Chemical Laboratory Coordination with CQAB Preparation of Daily Quality Control Reports Assist with sample packaging and shipping Preparation of report Layout of sample locations and building survey
Doug Ronk	Arrowhead	Sample Custodian	 Sample packaging and shipping COC documentation Sample labeling Final preparation of samples Layout of sample locations and building survey
Ben Williams Andy Arnold Jack Reasbeck Aaron Mathena	Arrowhead	Sampling Team	 Layout of sample locations and building survey Assist with sample collection Preparation of sample for off-site analysis

KEY PERSONNEL	ORGANIZATION	ROLE	RESPONSIBILITIES
Joe Dillon TBD			 Collection of field QA/QC samples Decontamination
Dan Mitchell	CENWK	Health & Safety Officer and QC Inspector	On-site H&S OversightQC Inspections
TBD	TBD		Concrete coring services
Diane Borthwick		Data Management	 Download of laboratory and field electronic data files into database Coordination with personnel involved with data validation, QCSR, and report preparation
Francis Zigmund	CENWK	Project Chemist	Chemistry oversight
Kurt Baer	CENWK	Project Engineer	 Technical oversight
Dan Mitchell	CENWK	Health & Safety	Health & Safety
TBD	TBD	Analytical laboratory for off-site analysis of PCBs and other parameters.	Chemical analysisLaboratory QA/QCRaw data summary report

Note: Any changes in personnel assignments are subject to CENWK approval.

Table 3-1
Summary of Sampling Depth Intervals by Selected Area

Media	Area of Concern	Sampling Intervals
Concrete	Flooring –1 st Floor Former	0 to 1 inch below concrete cap, and
	Process Areas	2 to 3 inches below concrete cap
Concrete	Flooring –1 st Floor Former	0 to 1 inch below concrete cap, and
	Traffic Areas	1 to 2 inches below concrete cap
Concrete	Flooring – 2 nd Floor Former	0 to 1 inch below concrete cap, and
	Process Areas	2 to 3 inches below concrete cap
Concrete	Flooring –2 nd Floor Former	0 to 1 inch below concrete cap, and
 	Traffic Areas	1 to 2 inches below concrete cap
Concrete	Columns in Basement	0 to 1 inch
Concrete	Former Areas of Transformers	0 to 1 inch below concrete cap, if
	and Motors	cap is present, and
		1 to 2 inches below concrete cap, if
		cap is present
Concrete	Miscellaneous oil-stained areas	0 to 1 inch below concrete cap, if
	on the first and second floors	cap is present, and
		1 to 2 inches below concrete cap, if
		cap is present
Concrete	Miscellaneous oil-stained areas	0 to 1 inch and 1 to 2 inches
	in the basement	
Waste	Chip Chute Area	0 to 2 feet
Waste	Catch Basins in Basement	0 to 1 feet and 2 to 3 feet
Concrete	Walls in Chip Chute Area	0 to 1 inch
Concrete (if	Flooring below waste pile in	0 to 1 inch
present)	Chip Chute Area	
Soil	Soil below flooring or directly	0 to 6 inches and 12 to 18 inches
	below waste pile in Chip Chute	
	Area	
Soil	Area outside Building and	0 to 6 inches and 12 to 18 inches
	Adjacent to Chip Chute Area	
Soil	Miscellaneous oil-stained areas	0 to 6 inches and 6 to 12 inches
	and non-oil-stained areas in the	
	basement	

Table 3-2
Summary of Areas of Concern for PCB Sampling and Estimated Number of Samples

Media	Area of Concern	Number of 20' X 20' Sections, if Applicable	Estimated Number of Sample Locations	Estimated Number of Samples	Estimated Area to be Investigated or Number of Columns
Concrete	Flooring –1 st Floor Former Process Areas	115	58 [115]	116 [230]	46,000 ft ²
Concrete	Flooring –1 st Floor Former Traffic Areas	92	47 [92]	94 [184]	36,800 ft ²
Concrete	Flooring – 2 nd Floor Former Process Areas	48	39 [78]	78 [96]	31,200 ft ²
Concrete	Flooring –2 nd Floor Former Traffic Areas	21	11 [21]	22 [42]	8,400 ft ²
Concrete	Columns in Basement	NA NA	30	30	30
Concrete	Former Areas of Transformers in the Basement	4	4	8	400 ft ²
Concrete	Former Areas of Motors in the Penthouse	4	4	8	3,200 ft ²
Concrete	Miscellaneous oil stained areas on first and second floor	NA	20	40	2,000 ft²
Concrete	Miscellaneous oil stained areas in basement	NA	30	60	3,000 ft ²
Waste	Chip Chute Area	NA	3	3	1,200 ft ²
Waste	Catch Basins in Basement	NA	4	8	400 ft ²
Concrete	Walls in the Chip Chute Area	NA	6	6	1,100 ft ²
Concrete (if present)	Flooring below Waste Pile in Chip Chute Area	NA	2	2	1,200 ft ²
Soil	Soil Below Flooring or directly below Waste Pile in Chip Chute Area	NA	2	4	1,200 ft ²
Soil	Area outside Building and Adjacent to Chip Chute Area	NA	12	24	1,350 ft ²
Soil	Miscellaneous oil-stained areas in basement	NA	30	60	3,000 ft ²
Soil	Miscellaneous non-oil- stained areas in basement	NA	4	8	400 ft ²

^{[] =} Estimated maximum number of sample locations assuming samples collected from 100% of the floor sectors; excludes additional perimeter sectors that may be sampled if results from planned sectors exceed the action level.

Table 3-3
Estimated Sampling Depths for Concrete Flooring by Selected Area

Media	Area of Concern	Sampling Depth ¹
Concrete	Flooring –1 st Floor Former	7 inches
	Process Areas	
Concrete	Flooring –1 st Floor Former	6 inches
	Traffic Areas	
Concrete	Flooring – 2 nd Floor Former	7 inches
	Process Areas	
Concrete	Flooring –2 nd Floor Former	6 inches
	Traffic Areas	
Concrete	Former Areas of Transformers	6 inches
	and Motors and in the	
	Miscellaneous Oil Stained	
	Areas	
Concrete	Miscellaneous Oil Stained	6 inches
	Areas in Traffic Areas	
Concrete	Miscellaneous Oil Stained	7 inches
	Areas in Process Areas	
Concrete	Miscellaneous Oil Stained	6 inches
	Areas in Basement	

The specified depth assumes that the cap thickness is 4 inches

Table 3-4
Summary of Methods for PCB Sampling

Media	Area of Concern	Sampling Method
Concrete	Flooring –1 st Floor Former Process Areas	Composite Concrete Sampling – Coring
Concrete	Flooring –1 st Floor Former Traffic Areas	Composite Concrete Sampling – Coring
Concrete	Flooring – 2 nd Floor Former Process Areas	Composite Concrete Sampling – Coring
Concrete	Flooring –2 nd Floor Former Traffic Areas	Composite Concrete Sampling – Coring
Concrete	Columns in Basement	Discrete Concrete Sampling - Drilling
Concrete	Former Areas of Transformers in the Basement	Discrete Concrete Sampling - Drilling
Concrete	Former Areas of Motors in the Penthouse	Discrete Concrete Sampling - Drilling
Concrete	Miscellaneous oil stained areas on first and second floor	Composite Concrete Sampling – Coring
Concrete	Miscellaneous oil stained areas in basement	Discrete Concrete Sampling - Drilling
Waste	Chip Chute Area	Waste Grab Sampling
Waste	Catch Basins in Basement	Waste Grab Sampling
Concrete	Walls in the Chip Chute Area	Discrete Concrete Sampling – Drilling
Concrete (if present)	Flooring below Waste Pile in Chip Chute Area	Discrete Concrete Sampling – Drilling
Soil	Soil Below Flooring or directly below Waste Pile in Chip Chute Area	Discrete Soil Sampling
Soil	Area outside Building and Adjacent to Chip Chute Area	Discrete Soil Sampling
Soil	Miscellaneous oil-stained areas in basement	Discrete Soil Sampling
Soil	Miscellaneous non-oil-stained areas in basement	Discrete Soil Sampling

Note: Refer to Sections 4.3.1 through 4.3.5 for specific procedures associated with each of the sampling methods.

Table 3-5
Remediation Waste Characterization Sampling Approach and Sample Quantities

Material Type	Sampling Approach	Estimated Sample Quantity		
Concrete	One sample will be collected from a pre-selected sector of the first floor that is likely to be removed during subsequent TSCA waste remediation (>50 ppm PCBs). Sample to be a composite of cores from 0 to 1 inch within the selected sector.	1		
Waste	One sample from $0-2$ feet will be collected from the Chip Chute waste pile. One composite sample (from $0-1$ feet and $2-3$ feet) will be collected from material within one of the basement catch basins.	2		
Soil				

Note: The samples described above will be analyzed for TCLP SVOCs, TCLP Metals, and GRO/DRO.

Table 3-6
Health and Safety Characterization Sampling Approach and Sample Quantities

Material Type	Sampling Approach	Estimated Sample Quantity
Concrete	Samples will be collected from three pre-selected sectors of the first floor that is likely to be removed during subsequent TSCA waste remediation (>50 ppm PCBs). Additionally, samples will be collected from two preselected sectors of the first or second floor that will be removed during building demolition (<50 ppm PCBs).	5
Waste Pile	Waste Pile One sample from $0-2$ feet will be collected from the Chip Chute waste pile. One composite sample (from $0-1$ feet and $2-3$ feet) will be collected from material within one of the basement catch basins.	
Soil	One sample will be collected from the soil outside Building 3 (adjacent to the Chip Chute area). One sample will be collected from an oil-stained portion of the basement floor. Additionally, one sample will be collected from a non-oil stained portion of the basement floor. Each sample will be composited from the $0-6$ inch and $12-18$ inch depth intervals.	3

Note: The samples described above will be analyzed for Total SVOCs and Total Metals.

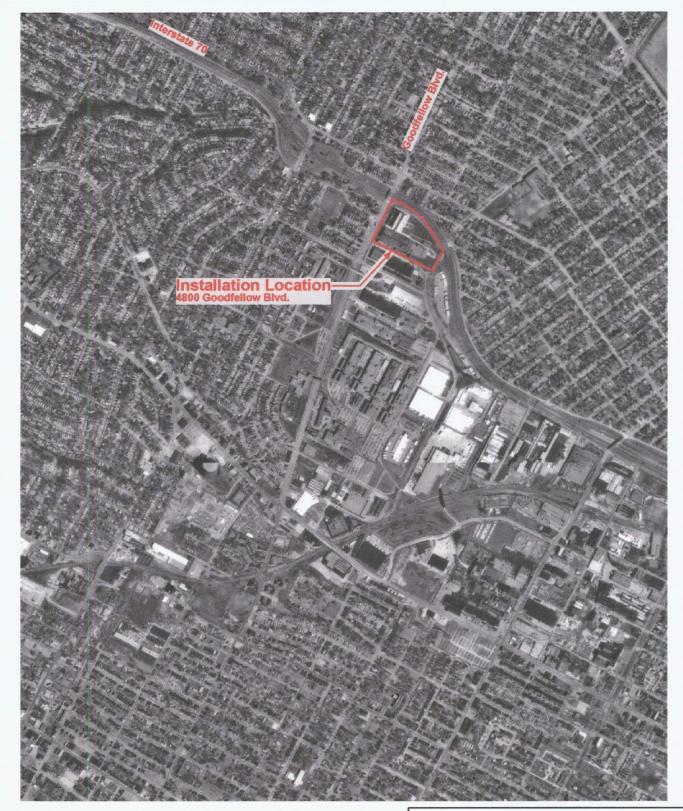
Table 4-1
Composite Sample Locations - First Floor
Building 3, St. Louis Army Ammunition Plant (SLAAP)
St. Louis, Missouri

e man	A MATERIAL PROPERTY.	LENGTH ENTERNA	A CONTRACTOR OF STREET	And the English	Area of Con	cern	State and the state	Cara Sala, mesa	CAN DELENS THE TO	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	
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CF-A05	CF-B03	CF1-C03	CF1-H23	CF1-K09	CF1-B06	CF1-D08	CF1-E08	CF1-F08	CF1-G08	CF1-H08	CF1-J08
CF-A11	CF-B04	CF1-D03	CF1-H24	CF1-K10	CF1-B07	CF1-D09	CF1-E09	CF1-F09	CF1-G09	CF1-H09	CF1-J09
CF-A12	CF-B05	CF1-E03	CF1-H28	CF1-K11	CF1-B08	CF1-D10	CF1-E10	CF1-F10	CF1-G10	CF1-H10	CF1-J10
CF-A13	CF-B09	CF1-F03	CF1-H29	CF1-K12	CF1-C08	CF1-D11	CF1-E11	CF1-F11	CF1-G11	CF1-H11	CF1-J1
CF-A14	CF-B10	CF1-F28	CF1-H30	CF1-K13	CF1-C09	CF1-D12	CF1-E12	CF1-F12	CF1-G12	CF1-H12	CF1-J1:
CF-A15	CF-B15	CF1-F29	CF1-H31	CF1-K14	CF1-C10	CF1-D13	CF1-E13	CF1-F13	CF1-G13	CF1-H13	CF1-J1
CF-A16	CF-B16	CF1-F30	CF1-H32	CF1-K15	CF1-C11	CF1-D14	CF1-E14	CF1-F14	CF1-G14	CF1-H14	CF1-J1-
CF-A18	CF-B17	CF1-F31	CF1-H33	CF1-K16	CF1-C12	CF1-D15	CF1-E15	CF1-F15	CF1-G15	CF1-H15	CF1-J1
CF-A19	CF-B18	CF1-F32	CF1-H34	CF1-K17	CF1-C13	CF1-D16	CF1-E16	CF1-F16	CF1-G16	CF1-H16	CF1-J1
CF-A20	CF-B19	CF1-F33	CF1-H35	CF1-K18	CF1-C14	CF1-D17	CF1-E17	CF1-F17	CF1-G17	CF1-H17	CF1-J1
CF-A21	CF-B20	CF1-G03	CF1-H36	CF1-K19	CF1-C15	CF1-D18	CF1-E18	CF1-F18	CF1-G18	CF1-H18	CF1-J1
CF-A22	CF-B21	CF1-G23	CF1-H37	CF1-K21	CF1-C16	CF1-D19	CF1-E19	CF1-F19	CF1-G19	CF1-H19	CF1-J1
CF-A23	CF-B23	CF1-G24	CF1-H38	CF1-K22	CF1-C17	CF1-D22	CF1-E22	CF1-F22	CF1-G22	CF1-H22	CF1-J1
CF-A24	CF-B24	CF1-G32	CF1-J23	CF1-K23	CF1-C18	CF1-D23	CF1-E23	CF1-F23	CF1-G25	CF1-H25	CF1-J22
CF-A25	CF-B25	CF1-G33	CF1-J24	CF1-K24	CF1-C19	CF1-D24	CF1-E24	CF1-F24	CF1-G27	CF 1-H23	
CF-A26	CF-B26		CF1-J25	CF1-K25	CF1-C22	CF1-D25	CF1-E25	CF1-F25	CF1-G28		CF1-K0
CF-A27	CF-B27	The second second	CF1-J34	CF1-K34	CF1-C23	OI I DZO	OI 1-L25	CF1-F27	CF1-G28		261
West of the State	CF-B28		CF1-J35	CF1-K35	CF1-C24			OF 1-F27	CF1-G29 CF1-G30		
			CF1-J36	CF1-K36	CF1-C25			100			
			CF1-J37	CF1-K37	3 020				CF1-G31		
		1.00	CF1-J38	CF1-K38							

Table 4-2
Composite Sample Locations - Scecond Floor
Building 3, St. Louis Army Ammunition Plant (SLAAP)
St. Louis, Missouri

Traffic Area		Area of Concern Process Area			
CF2-A18	CF2-B10	CF2-C10	CF2-D10	CF2-E10	CF2-F10
CF2-A19	CF2-B11	CF2-C11	CF2-D11	CF2-E11	CF2-F11
CF2-A20	CF2-B12	CF2-C12	CF2-D12	CF2-E12	CF2-F12
CF2-A21	CF2-B13	CF2-C13	CF2-D13	CF2-E13	CF2-F13
CF2-A22	CF2-B14	CF2-C14	CF2-D14	CF2-E14	CF2-F14
CF2-A23	CF2-B19	CF2-C18	CF2-D18	CF2-E18	CF2-F18
CF2-A24	CF2-B20	CF2-C19	CF2-D19	CF2-E19	CF2-F19
	CF2-B21	CF2-C22	CF2-D22	CF2-E22	CF2-F22
	CF2-B22	CF2-C23	CF2-D23	CF2-E23	CF2-F23
	CF2-B23	CF2-C24	CF2-D24	CF2-E24	CF2-F24
	CF2-B24	CF2-C25	CF2-D25	CF2-E25	CF2-F25
	CF2-B25				2,20
	CF2-B24		The same of the sa		

Figures





ST. LOUIS ARMY AMMUNITION PLANT ST. LOUIS, MISSOURI

FIGURE 1-1 SITE LOCATION MAP

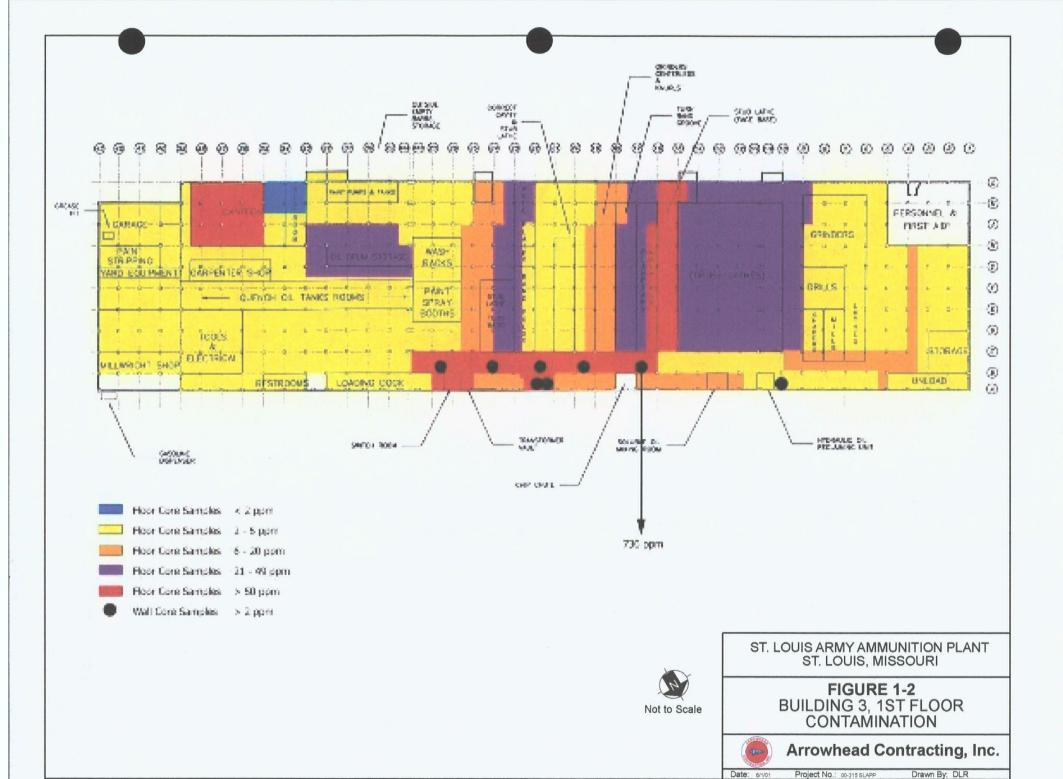


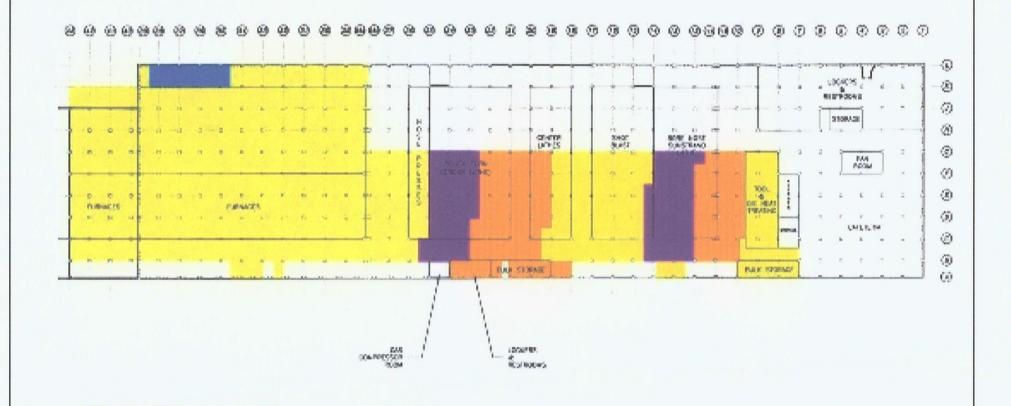
Arrowhead Contracting, Inc.

Source: USGS, Clayton, Missouri 7.5' x 15' Quadrangle aerial photography, flight date 1998.

ate: 6/01/01 Project No.: 00-215 SLAAP

Drawn By: DLF





Floor Core Samples < 2 ppm Floor Core Samples 2 - 5 ppm Floor Core Samples 6 - 20 ppm

Floor Core Samples 21 - 49 ppm

Floor Core Samples > 50 ppm



ST. LOUIS ARMY AMMUNITION PLANT ST. LOUIS, MISSOURI

FIGURE 1-3
BUILDING 3, 2ND FLOOR
CONTAMINATION

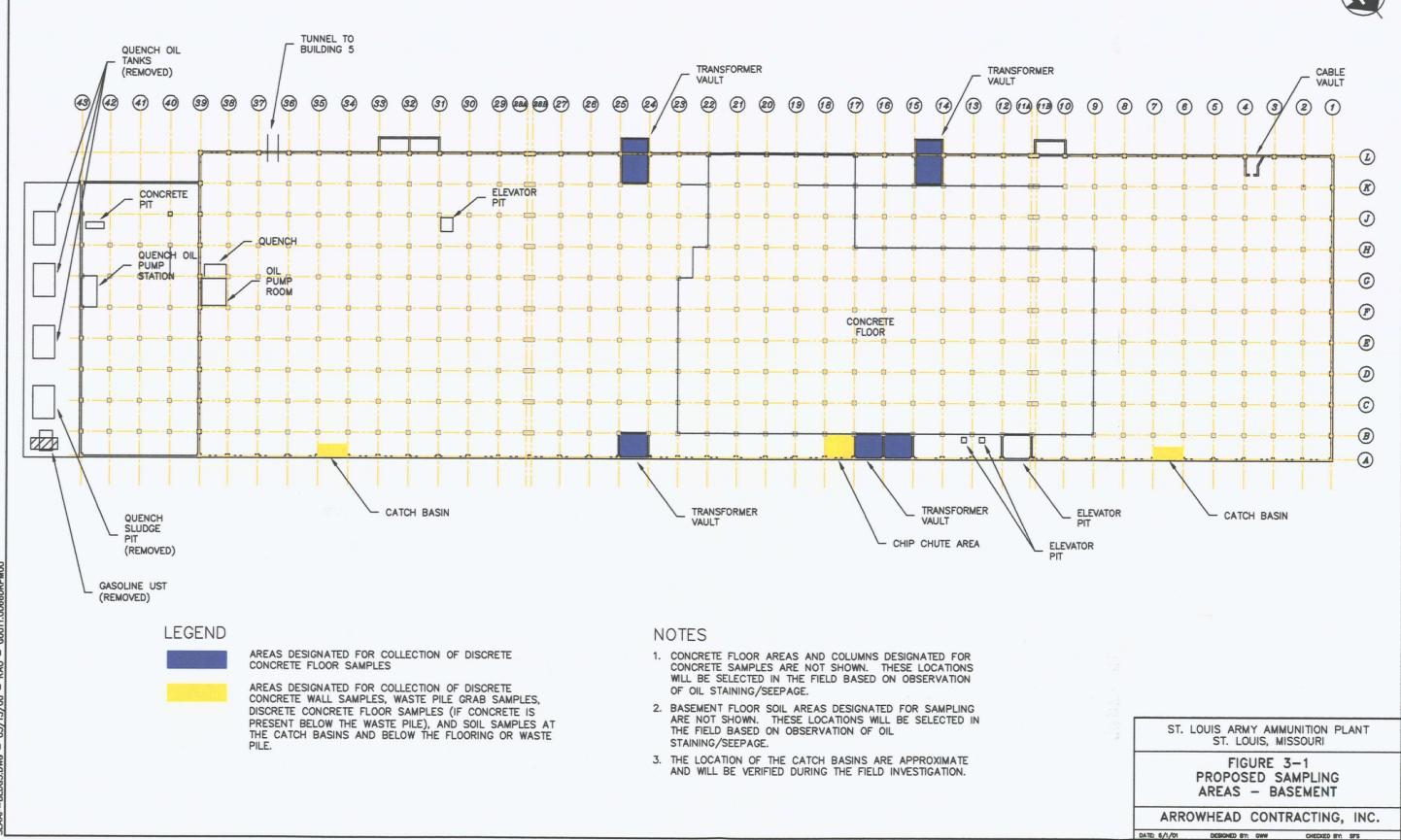


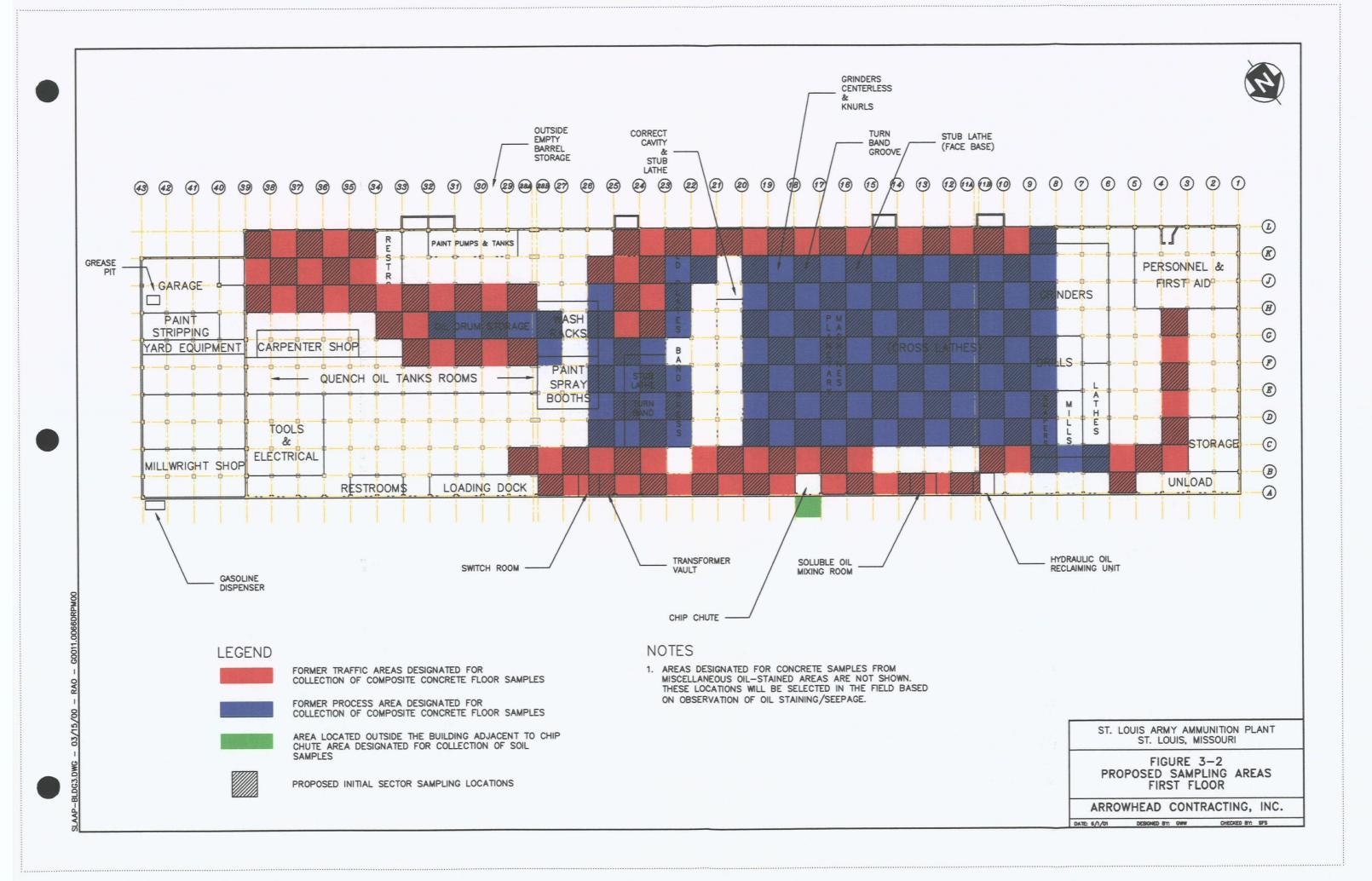
Arrowhead Contracting, Inc.

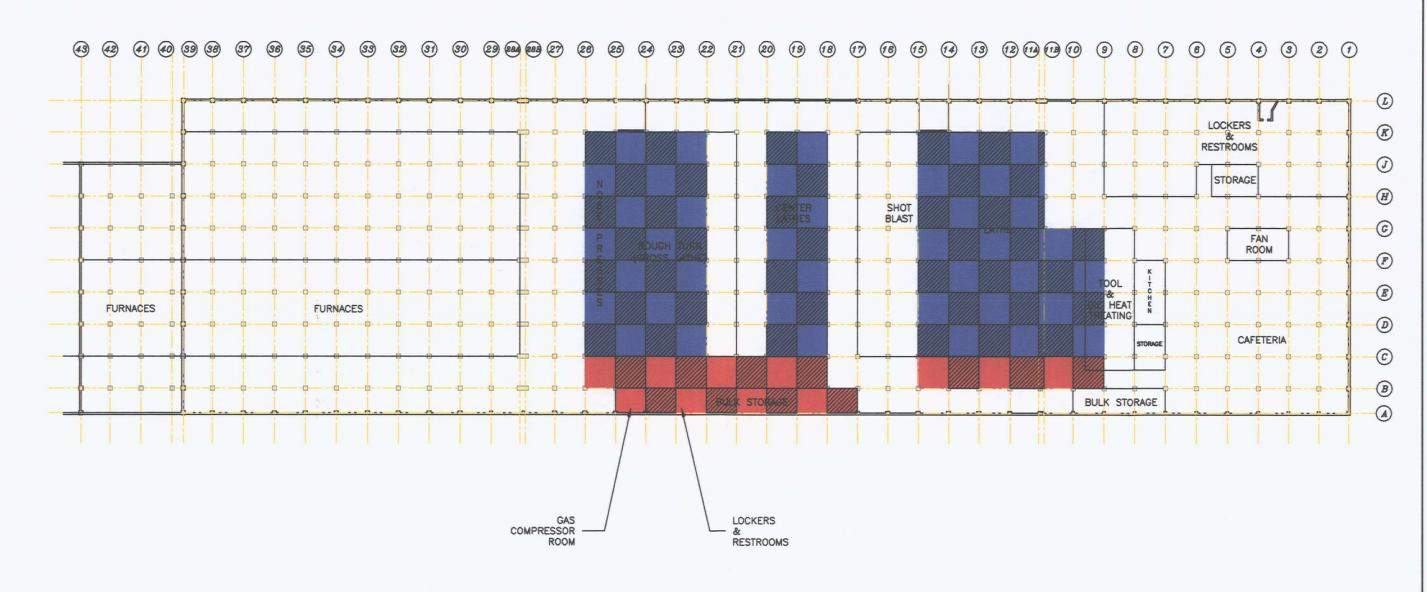
Date: 6/1/01 Project No.: 00-215 SLAPP

Drawn By: DLR









LEGEND

FORMER TRAFFIC AREAS DESIGNATED FOR COLLECTION OF COMPOSITE CONCRETE FLOOR SAMPLES FORMER PROCESS AREAS DESIGNATED FOR COLLECTION OF COMPOSITE CONCRETE FLOOR SAMPLES



PROPOSED INITIAL SECTOR SAMPLING LOCATIONS

NOTES

1. AREAS DESIGNATED FOR CONCRETE SAMPLES FROM MISCELLANEOUS OIL-STAINED AREAS ARE NOT SHOWN. THESE LOCATIONS WILL BE SELECTED IN THE FIELD BASED ON OBSERVATION OF OIL STAINING/SEEPAGE.

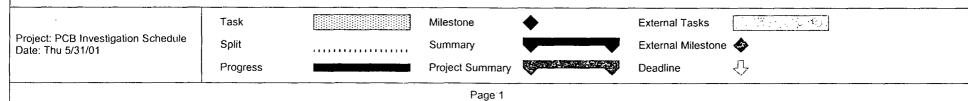
> ST. LOUIS ARMY AMMUNITION PLANT ST. LOUIS, MISSOURI

FIGURE 3-3 PROPOSED SAMPLING AREAS SECOND FLOOR

ARROWHEAD CONTRACTING, INC. DESIGNED BY: GWW CHECKED BY: SFS

Figure 10-1 Field Schedule **Determination of PCB TSCA Waste Quantities** St. Louis Army Amunition Plant

				<u> </u>							
ID	Task Name	Duration	Start	Finish	27, Jun 3, ' T S MW F						
1	Submit Final Sampling and Analysis Plan	1 day	Fri 6/1/01	·		ــــــــــــــــــــــــــــــــــــــ		13111	Olivilan	<u> </u>	1 (3 W)
2	Select Laboratory and Coring Service Contractor	1 day	Mon 6/4/01	Mon 6/4/01		:				:	i
3	CENWK Review of Consent Packages	7 days	Tue 6/5/01	Wed 6/13/01	*			:			
4	Procurement of Supplies and Equipment	10 days	Mon 6/4/01	Fri 6/15/01			Ъ			:	÷
5	Mobilize to Site from Kansas City	1 day	Sun 6/17/01	Sun 6/17/01	(-1-1-1-1-1-1-1	<u>#1-145#1#1#1#1</u>	T h		: 1	:	
6	Building Survey and Layout	2 days	Mon 6/18/01	Tue 6/19/01			Th.			•	
7	Collect Initial Concrete Floor Samples from First and Second Floors	8 days	Wed 6/20/01	Wed 6/27/01			Ť	Ш	:	:	
8	Collect Samples from Basement, Penthouse, and Outside Areas	2 days	Thu 6/28/01	Fri 6/29/01					<u>,</u>	:	,
9	Demobilize from the Site for the Holiday Break	1 day	Sat 6/30/01	Sat 6/30/01				F145			:
10	Mobilize Back to the Site	1 day	Thu 7/5/01	Thu 7/5/01						h :	•
11	Resume Collection of Concrete Floor Samples	6 days	Fri 7/6/01	Wed 7/11/01					· ·	Ť.	ь:
12	Resume Collection of Basement, Penthouse, and Outside Area Samples	4 days	Thu 7/12/01	Tue 7/17/01	1		:		:		
13	Demobilize from the Site	1 day	Wed 7/18/01	Wed 7/18/01	:	:					



Appendix A Project Forms



	The second secon	Sa	mple Collection Fiel	ld Sheet					
Project No:				Media: V	Vater				
Site:					Soil	-			
Sample Locat	tion:			Soi	Gas	_			
					Air	-			
Date:				Waste					
Time (Start):		Time (Finish	n):	Other					
Sample ID(s):				Sampling Personnel:					
Temp: F / C Wind: Calm Moderate High Precipitation: Rain: Lite Moderate Hea			High Heavy	Sampling Method/Equ	ip:	170			
Sample Depth	/Type/ID In	formation:	Same Company G. Market of the		section desired				
Depth	Composite	Time	Sample ID	Descr	iption				
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	YN					162			
	YN								
	YN								
	YN								
	YN								
Sample Conta					Sandy States of	1000			
Sample Co	ontainer	Preservative	Analysis Required	Method Number	Laborat	tory			
Notes/Sketch:									

					HTW	DRIL	LIN	G LOG			-	HOLE N	D.
1. COMF	ANY NAME					2. DI	RILLING	SUBCONTRACTOR				SHEET	
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3. PROJ	ECT				·······			4. LOCATION	··· -				
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J. NAME	O DIVILL	D.						6. MANUFACTURER	S DESIGNATION	OF DRILL			
7. SIZES DRILL	AND TYPE	S OF AMPLING		•				8. HOLE LOCATION					
EQUII	PMENT	•	-					9. SURFACE ELEVA	ΠON				
								10. DATE STARTED					
12. OVE	RBURDEN	THICKNESS		· - · · ·				15. DEPTH GROUND	WATER ENCOUN	TERED 11.	DATE C	OMPLETED	
13. DEP1	'H DRILLED	INTO ROCK						16. DEPTH TO WATE	R AND ELAPSED	TIME AFTE	R DRILL	ING COMPLETED	
14. TOTA	IL DEPTH C	OF HOLE						17. OTHER WATER L	EVEL MEASUREN	ENTS (SPE	CIFY		
											,		
18. GEO	FECHNICAL	SAMPLES	DI	STURBED	U	NDISTURBE)	19. TOTAL NUMBE	R OF CORE BOXE	S			
20. SAM ANA	PLES FOR YSIS	CHEMICAL		VOC	М	ETALS		OTHER (SPECIFY)	OTHER (SPE	PECIFY) OTH		HER (SPECIFY)	21. TOTAL CORE REC.
22. DISP	OSITION O	F HOLE	BA	CKFILLED	MONITO	RING WELL	٠,	OTHER (SPECIFY)	23. SIGNATURE	OF INSPEC	TOR		L%
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DEPTH b	DESCRIPTION OF MATERIALS	FIELD SCREENING RESULTS	GEOTECH SAMPLE OR CORE BOX NO.	ANALYTICAL SAMPLE NO.	BLOW COUNTS	REMARKS h			
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WINACTING!					Sheet	of		
Inspection Checklist - \	Waste	Pile G	rab S	ampling				
Location:	Date:				Photograph? If yes,			
	Time:				photograph num			
		_		-	Yes	Number		
Inspection Type (circle one): Initial		Da	aily QC		No			
Task	Yes	No	NA		Remarks			
Are all checklist items identified during the preparatory inspection being addressed?								
Are sampling locations being measured in reference to						The contract of the contract o		
existing features (columns, walls, etc.)?								
Is proper PPE in use?								
Is sampling equipment being decontaminated between each use?								
Are fresh gloves being used to handle each sample?								
Are waste material samples being collected from 0 to 2 feet?								
Is the material for each sample being thoroughly								
homogenized? Have the proper type and number of sampling containers been filled?								
Are sample containers being labeled properly?			\vdash					
Has the remaining sample volume been replaced into the holes?								
Are the samples being placed on ice?								
Has the chain-of-custody been completed for the samples?								
Has a sample collection field sheet been completed for each sample?								
Is all non-disposable equipment being decontaminated between each use?								
Has all IDW been properly contained?								
Did samplers record pertinent sampling information in the field								
log book? Were labels properly filled out (with sample ID, date, time,			-					
analyses, and preservative) and attached to all sample containers?								
Were samples properly packaged and custody tape placed on the shipping container?								
Were required QA/QC samples (duplicates, MS/MSD,								
rinsates) collected, packaged, and shipped to the laboratory								
along with the primary samples?								
Were QA/QC samples labeled to distinguish them from the corresponding primary sample?								
Were required split samples collected, packaged, and shipped								
to the USACE laboratory?								
Was the laboratory contacted the next day to confirm that the samples arrived at the laboratory in satisfactory condition?								
Notes:		•						



PACTINO						Sheet	of
Inspection Checklist - Disc	rete C	oncre	te Flo	or Samp	oling	11-07 1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-	
Location:	Date:				Photograph?	? If ves,	
	Time:				photograph		
Inspection Type (circle one): Initial		Da	aily QC	30	-		
Task	Yes	No	NA		Remar	rks	
Are checklist items identified during the preparatory inspection being addressed?							
Are coring locations being measured in reference to existing features (columns, walls, etc.)?						PROPERTY OF THE PROPERTY OF TH	
Is proper PPE in use?							
Is coring equipment being decontaminated between each use?							
Are fresh gloves being used to handle each sample?					Particular de la constantina della constantina d		
Are concrete cores being collected to a final depth of 2 inches below the concrete cap/concrete floor interface in former traffic areas (first and second floor), basement, tranformer vaults, and penthouse, or 3 inches below the concrete cap/concrete floor interface in former process areas (first and second floors)?							
Is the concrete saw being decontaminated between each use?							
Are the concrete core samples being cut into samples from 0"-1" and 1"-2" in former traffic areas (first and second floor), basement, tranformer vaults, and penthouse, and 0"-1" and 2"-3" in former process areas (first and second floors)?							
Is the drilling and sampling equipment being decontaminated between each use?							
Is the electronic balance been reset to zero (tared) before use?							
ls at least a thirty (30) gram sample being obtained for PCB analysis at each location?							
Are the sample containers being labeled properly?							
ls the remaining sample volume being retained in a separate container and labeled appropriately?							
Are the samples being placed on ice?							
Has the chain-of-custody been completed for the samples?							
Has a sample collection field sheet been completed for each sample?							
Is all non-disposable equipment been decontaminated between each use?							
Has all IDW been properly contained?							
Did samplers record pertinent sampling information in the field log book?							-
Were labels properly filled out (with sample ID, date, time, analyses, and preservative) and attached to all sample containers?							
Were samples properly packaged and custody tape placed on the shipping container?							
Were required QA/QC samples (duplicates, MS/MSD, rinsates) collected, packaged, and shipped to the laboratory along with the primary samples?							
Nere QA/QC samples labeled to distinguish them from the corresponding primary sample?							
Were required split samples collected, packaged, and shipped to the USACE aboratory?							
Nas the laboratory contacted the next day to confirm that the samples arrived at the laboratory in satisfactory condition?							
Votes:							



Daily QC	Photograph? If yes, photograph number. Yes Number No Remarks
<u> </u>	Yes Number
<u> </u>	No
o NA	Remarks
	I/CIIIdI NO



Sheet of Inspection Checklist - Composite Concrete Floor Sampling Location: Date: Photograph? If yes, Time: photograph number. Yes Number Inspection Type (circle one): Initial Daily QC Yes No NA Remarks Are checklist items identified during the preparatory inspection being addressed? Are coring locations being measured in reference to existing features (columns, walls, etc.)? Is proper PPE in use? Is coring equipment being decontaminated between each use? Are fresh gloves being used to handle each sample? Are concrete cores being collected from each quadrant, and to a final depth of 2 inches below the concrete cap/concrete floor interface in former traffic areas or 3 inches below the concrete cap/concrete floor interface in former process areas? Is the concrete saw being decontaminated between each use? Are the concrete cores from each quadrant being cut into samples from 0"-1" and 1"-2" in former traffic areas and 0"-1" and 2"-3" in former process areas? Is the drill and sampling collection equipment being decontaminated between each use? Is the electronic balance been reset to zero (tared) before use? Is a five (5) gram sample from each quadrant and depth interval being collected for PCB Are the sample containers for the respective sector and depth being labeled property? Is the remaining sample volume been retained in a separate container and labeled appropriately? Have the individual 5-gram aliquot samples from each quadrant and depth in each sector been adequately composited within the sample container for PCB analysis? Are the composited samples being placed on ice? Has the chain-of-custody been completed for the samples? Has a sample collection field sheet been completed for each sample? Is all non-disposable equipment being decontaminated before each use? Has all IDW been properly contained? Did samplers record pertinent sampling information in the field log book? Were labels properly filled out (with sample ID, date, time, analyses, and preservative) and attached to all sample containers? Were samples properly packaged and custody tape placed on the shipping container? Were required QA/QC samples (duplicates, MS/MSD, rinsates) collected, packaged, and shipped to the laboratory along with the primary samples? Were QA/QC samples labeled to distinguish them from the corresponding primary Were required split samples collected, packaged, and shipped to the USACE laboratory? Was the laboratory contacted the next day to confirm that the samples arrived at the laboratory in satisfactory condition? Notes:

Definable Feature of Work:				Contract No: DACW41-00-D0019 Task Order 0002				
Definable Feature of Work:			Inspection Date:					
Location: St. Louis Army Ammur	nition Plant		Specification: Sampling & Analysis Plan					
Requirements/Reference	Yes	No	N/A	Remarks				

			-		
	eparatory Inspection	,	~		
1.	Have field personnel reviewed the	ŀ			
1	Sampling and Analysis Plan (SAP) and				
<u></u>	associated procedures?	<u> </u>			
2.	Are project forms (i.e. DQCRs, NCRs,				+
1	FWVs, COCs, sample collection field	\	1		\
L	sheets) available?	<u> </u>	ļ		
3.	Has a project kick-off meeting occurred?	ł			
	Were H&S issues covered during the				
	meeting?				
4.	Have necessary utility clearances been	}			
	obtained?	<u> </u>	1		
5.	Has a source of electrical power been				
	established for field equipment?	L			
6.	Has a filing system for project evidence				
	files been established?			İ.	
7.	Has a sample staging/packaging area		ľ		
	been established?			<u> </u>	
8.	Has a subcontract been set-up with an				
	approved laboratory for off-site analytical		-		
<u> </u>	activities – PCBs, SVOCs, metals?				
9.	Has the analytical laboratory been notified		Ì		
	of the start of sampling activities?				
10.	Have sample containers (jars and bottles)		1		
ľ	been received in good condition? Do	ŀ			
	sample containers contain the proper]			
	preservatives?		<u> </u>		
11.	Have sample packaging supplies (i.e.	ŀ			
	plastic bags, coolers, tape) been received?		i		
12.	Have sampling personnel been trained on				
	the sample numbering system?				
13.	Has PPE been received in good				
	condition?				
14.	Has concrete coring, sawing, and drilling				
	equipment been received in good			İ	
	condition and checked for operation?				
15.	Has sampling equipment and supplies		1		
	(augers, bowls, etc.) in good condition			1	
	and clean?			<u> </u>	
16.	Has health and safety monitoring			ļ	
	equipment (i.e. dust, CO, noise, heat)		•		
	been received and checked for operation?			ļ	
	Has a "cool area" been established?			ļ	
18.	Has the Health & Safety Officer reviewed			1	
	the H&S Plan with field personnel? Have			l	

CONTRACTING IS		Contract No: DACW41-00-D0019 Task Order 0002				
Definable Feature of Work:			Inspection Date:			
Location: St. Louis Army Ammunition Pla	ant .		Specifi	cation: Sampling & Analysis Plan		
Requirements/Reference	Yes	No	N/A	Remarks		
Preparatory Inspection	,					
field personnel signed the H&S Plan compliance certification?						
19. Have MSDSs (or other safety information) been received for potential contaminants to be encountered (i.e. PCBs) and chemical products to be used during the project?						
20. Have containers for IDW management been received in good condition?						
21. Has deionized water, Alconox, and methanol for equipment decontamination been received?						
22. Have decontamination and IDW disposal procedures been reviewed with field personnel?						

Date:



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	Nonconfo	rmance R	Report		
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Nonconformance Description (include spec	cific requirement violated):				
	Identified By:			Date:	
Root Cause of Nonconforming Condition:					
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Corrective Action to be Taken (include date	when action(s) will completed):				
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Action to be Taken to Preclude Recurrence:					
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Project Mana	ager		QA Officer		
Corrective Action Completed By:		Verification Co	ompleted By:		
Date:		Date:			



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	USACE Site Lead		Date:	•		
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Approved By:						
	USACE Technical Manager	-	Date:			
Approved By:						
	JSACE Contracting Officer's Repre-	sentative	Date:			
,	John Contracting Officer's Repre	Scritative	Date.			- 1



DAILY QUALITY CONTROL REPORT

REPORT NO CONTRACT NO. <u>DACW41-00-D0019</u> Date:
ATTN:
LOCATION OF WORK: St. Louis Army Ammunition Plant (SLAAP), Building 3
DESCRIPTION OF WORK: Concrete and Soil Sampling
WEATHER:
1. Work Performed:
1. Work renormed.
2. Tests Performed and Results:
3. Verbal Instructions Received:
4. Corrective Actions Proposed/Taken:
5 Health and Cafaty James
5. Health and Safety Issues:
6. Other Comments:

7. CERTIFICATION: I certify that the above report is complete and correct and that I, or my authorized representative, have inspected all work performed this day by the contractor and each subcontractor and have determined that all materials, equipment, and workmanship are in strict compliance with the plans and specifications, except as may be noted above

Arrowhead Contracting, Inc. QA/QC Manager



Company	Constitution and the second			Sr	neet	of
C	orrective	Action R	Request			100
CAR Number:	Project Numbe	er:			Date:	
Discrepancy (include specific requirements violated):						
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Corrective Action Taken/Proposed to Correct Discrepancy:						
Corrective Action Taken to Prevent Reoccurrence (including the	he cause of the	e discrepancy	<i>(</i>):			
Cost/Schedule Impact:						
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Corrective Action Taken By:		Date When	Corrective Action Will Be Com	pleted:		
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Corrective Action Evaluated:		Verification of	of Implementation:			
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CC: Distribtution						
Approved By:						
Arrowhead Project Manager		•	Date:	_		
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Arrowhead Quality Control Mana	ger		Date:			
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USACE Technical Manager		!	Date:	-		
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List of Acronyms

AA atomic absorption

ASTM American Society for Testing and Materials

CENWK Kansas City District Office of the U.S. Army Corps of Engineers Northwest

Division

CFR Code of Federal Regulation
CLP Contract Laboratory Program

COC chain-of-custody

DQCR Daily Quality Control Report

DQI data quality indicator
DQO data quality objective
DRO diesel range organics
ECD electron capture detector

ELCD electrolytic conductivity detector EPA Environmental Protection Agency

FADL Field Activity Daily Log
FSP Field Sampling Plan
FWV Field Work Variance

g gram

GC gas chromatography

GC/MS gas chromatography/mass spectroscopy

GRO gasoline range organics

HNO₃ nitric acid

HPLC high-performance liquid chromatography
HTRW Hazardous, Toxic, and Radioactive Waste

ICP inductively coupled plasma

ICPAES Inductively Coupled Plasma Atomic Emission Spectroscopy

LCS laboratory control sample

LIMS Laboratory Information Management System

LOR Letter-of-Receipt
MDL method detection limit
mg/kg milligrams per kilogram
mg/L milligrams per liter

mL milliliter
MS matrix spike

MSD matrix spike duplicate NCR Nonconformance Report

NIST National Institute of Standards and Technology

ppm parts per million

List of Acronyms (continued)_____

QA quality assurance

QAPP Quality Assurance Project Plan

QC quality control

QCSR Quality Control Summary Report

RCRA Resource Conservation and Recovery Act

RL reporting limit

RPD relative percent difference SAP Sampling and Analysis Plan

SLAAP St. Louis Army Ammunition Plant

SOP standard operating procedure SVOC semivolatile organic compound

TCLP Toxicity Characteristic Leachate Procedure

TPP technical project planning
USACE U.S. Army Corps of Engineers

XRD X-ray diffraction

μg/kg microgram(s) per kilogram μg/L microgram(s) per liter

1.0 Introduction

This portion (Part II) of the Sampling and Analysis Plan (SAP) consists of the Quality Assurance Project Plan (QAPP). The QAPP will be used to guide analytical and quality assurance/quality control (QA/QC) activities during field work at Building 3 at the Saint Louis Army Ammunition Plant (SLAAP) (refer to Figure 1-1 of the FSP for the location of SLAAP). The United States Army Corps of Engineers (USACE) and the United States Environmental Protection Agency (EPA) require participation in a centrally managed quality assurance (QA) program for environmental monitoring efforts. Any party generating data for an environmental monitoring project has the responsibility to implement procedures to ensure that the data is of adequate quality (in terms of precision, accuracy, representativeness, and completeness) and that the data is appropriately documented. To ensure these responsibilities are met, parities involved in the project must adhere to the requirements specified in this QAPP.

The Field Sampling Plan (FSP) portion (Part I) of this SAP contains detailed descriptions of, among other things, the site layout and history, project scope and objectives, planned sampling activities, sampling rationale, number of samples, and sampling methods. This QAPP (Part II of the SAP) presents a detailed discussion of the analytical and QA/QC activities associated with the Building 3 sampling effort, including data quality objectives, analytical methods, field QA/QC sampling, laboratory QC checks, laboratory calibration procedures, and data validation and reporting. Despite covering different aspects of the project, the contents of each plan are not mutually exclusive. It is intended that the QAPP and FSP be used jointly for purposes of project management.

It should be noted that analytical activities and methodologies associated with analysis of QA split samples to be performed by USACE at a USACE-designated laboratory are not addressed within this document. This QAPP applies to Contractor analytical requirements only. However, the collection of the QA split samples by the Contractor is addressed herein.

The QAPP has been organized into sixteen sections. The contents of each section are summarized below:

- Section 1.0 Introduction
 - Discusses the general purpose and rationale for development of the QAPP and the relationship of the QAPP to the FSP.

- Section 2.0 Project Organization and Responsibilities
 - Presents the project organization and responsibilities as they relate to analytical services.
- Section 3.0 Data Quality Objectives
 - Presents, in general terms, the data quality design process and selection of quality objectives for project data.
- Section 4.0 Sampling and Analysis Program
 - Presents the type of samples to be collected and the corresponding analyses to be performed.
- Section 5.0 Sample Containers, Preservation, and Holding Times
 - Presents the requirements for sample containers, preservation, and holding times.
- Section 6.0 Field QA/QC Samples
 - Presents the types QA/QC samples to be collected during the project, including the frequency of collection.
- Section 7.0 Analytical Methods
 - Presents a general description of the analytical methods and sample preparation procedures.
- Section 8.0 Laboratory Calibration Procedures
 - Presents the general procedures for maintaining the accuracy of instruments and equipment used for conducting laboratory analyses.
- Section 9.0 Laboratory QA/QC Checks
 - Presents details regarding the types of QA/QC samples that will be analyzed to check the performance of the laboratory.
- Section 10.0 Laboratory Preventative Maintenance
 - Presents a general description of preventative maintenance associated with laboratory instruments and equipment.
- Section 11.0 Analytical Corrective Actions
 - Presents the corrective actions that will be implemented in the event problems are encountered with analytical equipment or data quality criteria.
- Section 12.0 Calculation of Data Quality Indicators
 - Presents general descriptions of the methods for assessing project data relative to data quality indicators, including accuracy, precision, completeness and comparability.
- Section 13.0 Data Reduction, Validation, and Reporting

- Presents a description of the overall data review process to ensure the validity and usability of project data.
- Section 14.0 Performance and System Audits
 - Presents a description of the audits that will be conducted to ensure that analytical and QA/QC activities are conducted in accordance with the QAPP.
- Section 15.0 Quality Assurance Reports to Management
 - Presents details regarding the various types of quality assurance reports that will be prepared and submitted to management during the project.
- Section 13.0 References
 - Presents a list of references associated with this QAPP.

All QA/QC procedures will be in accordance with applicable professional technical standards, EPA and USACE requirements, government regulations and guidelines, and specific project goals and requirements. This following are the primary references used for the development of this QAPP:

- Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans (EPA 1991)
- EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations (EPA 1994a)
- Chemical Data Quality Management for Hazardous, Toxic, Radioactive Waste Remedial Activities (USACE 1998)
- Requirements for the Preparation of Sampling Analysis Plans (USACE 1994a).

2.0 Analytical Organization and Responsibilities

The general project organization and responsibilities are presented in Chapter 2.0 of the FSP (referencing Table 2-1). The table lists the CENWK, Contractor, and subcontractor positions that have responsibility for obtaining analytical data for the project. The information presented in this section, provides the organization and responsibilities of the Contractor environmental laboratory(ies) that will provide analytical services under the contract.

Analytical laboratory support specific to the Building 3 sampling effort will be obtained from an independent chemical laboratory. The selected subcontract laboratory shall be validated by USACE Center of Expertise. Relevant QA Manuals, laboratory qualification statements, certifications, and license documentation will be made available upon request.

Organization charts outlining the key laboratory personnel and organization will be identified in the QA Plans submitted by the laboratory. The responsibilities of key personnel will also be described in the QA plan. Key analytical personnel include:

- Quality Assurance/Quality Control Manager
- Project Manager
- Laboratory Manager
- Laboratory Technicians and Sample Custodians
- Data Manager

Note: Prior to commencement of field activities for the project, the Contractor will provide a complete copy of the SAP to the subcontract laboratory.

3.0 Data Quality Objectives

Data Quality Objectives (DQOs) are qualitative and quantitative statements derived from the DQO process that specify, from an end users perspective, the quality of data required to support decisions made during investigative activities. The DQOs specify the maximum level of uncertainty the user is willing to accept in order to accurately make project decisions. DQOs are developed prior to data collection and should be specified for all data collection activities that take place.

3.1 Project Objectives

The underlying objective with respect to data quality is to generate data that is technically sound and legally defensible. In terms of the Building 3 sampling effort, the specific objectives are to:

- Identify areas and quantities of contamination in Building 3 that will be included in a subsequent remedial action.
- Verify that oil staining is a reliable indicator for identifying PCB contamination in basement soils.
- Pre-determine the waste characteristics of Building 3 concrete, waste material (Chip Chute Waste pile and catch basins), and soil (basement floor and outside adjacent to Chip Chute) for potential removal and off-site disposal during a subsequent remedial action.
- Pre-assess the health and safety concerns (i.e. personnel exposure) associated with Building 3 concrete, waste, and soils to support planning for a subsequent remedial action and building demolition.
- Assess personnel exposure to silica from potential dust-generating activities during the Building 3 sampling effort
- Characterize investigation-derived waste (IDW) (decontamination water) from the Building 3 sampling effort to determine proper disposal methods.

This is to be accomplished through the proper implementation of the field sampling procedures, chain of custody (COC) documentation, controlled laboratory analysis, and validation of the reported data prior to their use. The necessary procedures for field sampling and COC are discussed in the FSP. Procedures for laboratory analysis and data validation are discussed in other sections of this QAPP.

3.2 Data Quality Design Process

As described in the USACE Engineering Manual, EM 200-1-2, *Technical Project Planning* (TPP) Process (USACE 1998), the data quality design process is basically a four-phase process performed to identify the data needed to support specific project decisions and to create a data

collection program to collect the necessary data. The DQOs generated as a result of the TPP process are project-specific statements that incorporate nine data quality requirements:

- 1. Project objective(s) satisfied
- 2. Data user perspective(s) satisfied
- 3. Contaminant or characteristic of interest identified
- 4. Media of interest identified
- 5. Required sampling areas or locations and depths identified
- 6. Number of samples required
- 7. Reference concentration of interest or other performance criteria identified
- 8. Sampling method identified
- 9. Analytical method identified

Most of these requirements are addressed in Section 3.0 of the FSP. The remaining requirements are addressed in this QAPP. A general summary of the DQO design process for the Building 3 project is presented in Table 3-1.

3.2.1 Identify Current Project Strategy

The first phase of the TPP process brings together decision-makers and technical personnel (e.g., customer, data users, and regulators) to identify an overall strategy to manage a site from its current condition to the desired closeout condition. Integral to development of a strategy for the site is establishing both short- and long-term objectives for the project. These objectives are the driver for collecting data. The overall strategy for the Building 3 project is discussed in detail in the FSP. Project objectives are presented in Section 3.1.

3.2.2 Determine Data Needs

Following establishment of the project strategy and objectives, data needs are identified commensurate with the expectations of the end-users of the data, such that the level of data quality will satisfy all project objectives. During this phase, technical personnel evaluate existing data, if any, and define the media-type, chemical requirements and numbers of samples necessary to statistically support the data users decision making process. Considerations include:

- Data needed to satisfy project objectives
- Data user
- Intended use of data
- Number of samples necessary to satisfy intended use
- Reference concentration of analyte of interest
- Area of interest or desired sampling location(s) and depth(s).

The data needs for the Building 3 project, including the areas of interest (concern), sampling locations, sample depths, and types and number of samples, is presented in Section 3.0 of the FSP (referencing Tables 3-1 through 3-4). Table 3-1 of the QAPP summarizes the data needs and presents the analytes of interest for the project.

3.2.3 Develop Data Collection Options

The next phase of the TPP is to design and plan the sampling and analysis activities necessary to fulfill the data needs. During this phase, the collection options are developed. Technical personnel document the requirements for data collection options, including the appropriate sampling and analysis methods. The documentation process must include:

- Data needs being met
- Project objectives to be satisfied
- Number of samples are to be collected
- Locations from where the samples are to be collected
- Sample collection methods to be used
- Sample analysis methods to be used
- List limitations, benefits or requirements associated with each data collection option.

This phase of the DQO design process was discussed in detail in Sections 3.0 and 4.0 of the FSP.

3.2.4 Finalize Data Collection Program

This final phase is to create a data collection program that best fits the short-term and long-term objectives. The design of the data collection program is performed by the PM, key data users, and data implementors and should include the regulators and stakeholders to ensure representation of all key data needs. The type and frequency of samples to be collected, as well as definition with respect to the data collection options will be identified during this phase. Additionally, project-oriented DQO statements are prepared that describe the intended data use(s), the data need requirements, and the means to achieve them. Table 3-1 presents the DQO statements for the Building 3 project. The overall sampling and analysis program resulting from the DQO design process is discussed in Section 4.0.

3.3 Quality Assurance Objectives for Analytical Data

The final step in establishing the DQOs is to determine the analytical data quality indicators (DQIs). The primary DQIs include precision, accuracy, completeness, sensitivity,

representativeness, and comparability. The laboratory chosen to perform the analytical work will provide their laboratory quality assurance plan, which shall include the Standard Operating Procedures (SOPs) and laboratory-specific quality control limits for all contracted parameters. Based on the SOPs, the Contractor shall ensure that the laboratory is capable of complying with project-specific DQIs. A detailed discussion of the methods for calculating the primary DQI parameters is found in Section 12.0 of this QAPP. The DQI parameters are defined as follows:

- **Precision** Precision is determined and reported as the relative percent difference (RPD) between the results for field duplicates and/or between the results for matrix spike/matrix spike duplicate (MS/MSD) samples. Data with acceptable quality shall meet the precision criteria presented in Tables 7-2, 7-4, 7-6, and 7-7.
- **Accuracy** Accuracy is determined and reported as the percent recovery from the analysis of a reference material, MS/MSD, and /or laboratory control sample (LCS). Data with acceptable quality shall meet the accuracy criteria presented in Tables 7-2. 7-4. 7-6, and 7-7.
- Completeness Completeness is determined for separate but integrated functions.
 - Sample Collection Completeness is calculated by comparing the number of samples actually collected in the field to the number of samples planned to be collected. Acceptance criteria for sample collection completeness shall be 95%.
 - Acceptable Data Completeness is defined as the percentage of useable data versus the total amount of data generated. Acceptable data are generated following a review (validation) of the data using the analytical method criteria (SW-846). Acceptable data are all data which have completed the review or validation process and have not been rejected. Acceptance criteria for acceptable data completeness shall be 95% for each analytical method defined in this QAPP.
 - Quality Data Completeness is defined as the percentage of quality data versus the total set of data. Quality data are analytical data obtained from a sample delivery group which meet all batch quality control criteria. Completeness criteria for quality data shall be 80%.
- Sensitivity is a quantitative reflection of the method detection limit (MDL). The reporting limit (RL) (also referred to as practical quantitation limit) is a secondary indicator of sensitivity. The MDLs and RLs are calculated by the analytical laboratory in accordance with 40 CFR Part 136 Appendix B. The RLs for the analytical methods to be used for this project are presented in Tables 7-1, 7-3, 7-5, 7-7, and 7-8. The subcontract laboratory shall submit SOPs identifying the MDLs for each analytical method.
- Representativeness/Comparability Representativeness and comparability are both qualitative statements about the data which can provide quality data if the sampling set is adequately prepared and standard method of analysis are used for chemical analysis.

4.0 Sampling and Analysis Program

Based on the DQO design process discussed in Section 3.0, a project-specific sampling and analysis program was developed and is summarized in Table 4-1. The sampling effort performed at Building 3 will involve collection of samples for the following purposes consistent with the project objectives:

- Samples collected for PCB identification (quantity and volume estimates)
- Samples collected for verification of oil-staining as a selection criteria for PCB contamination
- Samples collected for remediation waste pre-determination
- Samples collected for health and safety pre-assessment
- Samples collected for health and safety monitoring (i.e. silica)
- Samples collected for IDW characterization

This sampling program will involve the collection of samples from the following media type:

- Concrete
- Soil
- Waste material
- IDW water samples
- Filter cassettes (air monitoring)

Areas of the Building 3 to be sampled are identified on Figures 3-1, 3-2, and 3-3 of the FSP. The rationale for the selection of these areas is discussed in detail in Section 3.0 of the FSP. Sampling methods are discussed in Section 4.0 of the FSP. Estimates of the number of samples to be collected by media type are presented in Tables 4-1 and 6-2. Additional portions of select samples will be collected to meet QA/QC requirements, including duplicates, QA split samples, and field blanks as discussed in Section 6.0. The collection frequencies for field QA/QC samples are presented in Table 6-1. Estimates of the number of QA/QC samples to be collected are presented in Table 6-2.

Samples will be analyzed for the following parameters:

- PCBs
- Total Metals
- Total semivolatile organic compounds (SVOCs)
- Toxicity Characteristic Leaching Procedure (TCLP) Metals

- TCLP SVOCs
- Gasoline range organics (GROs) and diesel range organics (DROs)
- Crystalline silica

The SW-846 methods that will be used to analyze samples for these parameters (excluding silica) are presented in Table 4-1, and are discussed in further detail in Section 7.0. Sample container, sample volume, preservation and holding time requirements for the analytical parameters are discussed in Section 5.0 and presented in Tables 5-1, 5-2, and 5-3.

Filter cassettes from air monitoring activities will be analyzed for crystalline silica using NIOSH Method 7500. For reference and use by the subcontractor laboratory, a copy of NIOSH Methods 0600 and 7500 for total respirable dust and silica analyses, respectively, is provided in Appendix A. The remainder of this QAPP applies primarily to analysis site samples by SW-846 methods. Further details regarding health and safety monitoring are discussed the Safety, Health and Emergency Response Plan (SHERP).

5.0 Sample Containers, Preservation, and Holding Times

Sample containers, chemical preservation techniques, and holding times for concrete, soil, waste pile, and water samples collected during the Building 3 sampling effort are presented in Tables 5-1, 5-2, and 5-3. The specific number of containers required for this study will be estimated and supplied by the subcontracted analytical laboratory. When required by the analytical laboratory, additional sample volumes will be collected and provided for laboratory QC samples (laboratory duplicates, MS/MSD).

All sample containers will be provided by the analytical laboratory, which will also provide the required types and volumes of preservatives for the sample containers. Temperature preservation will be maintained at 4 C (±2 C) immediately after collection and will be maintained within this temperature range until the samples are analyzed. In the event that sample integrity, such as holding times, cooler temperatures, etc., is compromised, re-sampling will occur as directed by the CENWK Project Manager. Any affected data will be flagged and qualified per data validation instructions and guidance.

6.0 Field QA/QC Samples

Quality assurance/quality control (QA/QC) samples are analyzed for the purpose of assessing the quality of the sampling effort and of the reported analytical data. QA/QC samples to be used for the Building 3 project include field duplicates, USACE split samples, equipment rinsate blanks, and MS/MSD samples. Table 6-1 presents the frequencies at which the samples will be collected and analyzed. Table 6-2 presents the estimated numbers of QA/QC samples to be collected during the project.

6.1 Field Duplicates

These samples are collected by the sampling team for analysis by the subcontractor laboratory. The purpose of these samples is to provide site-specific, field-originated information regarding the homogeneity of the sampled matrix and the consistency of the sampling effort. These samples are collected concurrently with the primary samples at the same time and location. Duplicate samples will be collected from each media type and submitted to the subcontractor laboratory for analysis. Duplicates will be collected at a frequency of 10% of the total planned field samples.

6.2 USACE Split Samples

These samples are collected by the sampling team and sent to a USACE QA laboratory for analysis. Split samples provide an independent assessment of the subcontractor laboratory performance. The Contractor will coordinate with the designated QA laboratory not less than 48 hours before sampling to ensure that the laboratory is alerted to receive the QA samples and process them within required holding times. Split samples will be collected from the same sample as the field duplicate (Section 6.1) at frequency of 10% of the total planned field samples.

6.3 MS/MSD Samples

MS and MSD project samples that are "spiked" by laboratory with known quantities of analytes. The spiked samples are then and subjected to the entire analytical procedure. The MS is used to verify the accuracy of the analytical method (for a particular matrix) by measuring percent recovery of the analyte. The MSD is used to assess the precision of the analytical method. To meet MS/MSD requirements, the laboratory typically needs additional volume of the sample collected in the field. If requested by the laboratory, MS/MSD samples will be collected at a frequency of 20% of the total planned field samples.

6.4 Equipment Rinsate Blanks

These samples will be taken from the water rinsate collected during equipment decontamination activities. Rinsate blank samples will consist of "clean" (analyte-free) water used as a final rinse of decontaminated sampling equipment. They will be collected and submitted for analysis of the parameters of interest. Equipment rinsate blanks are used to assess the effectiveness of the decontamination process, the potential for cross contamination between sampling locations, and incidental field contamination. Equipment rinsate blanks will be collected at a frequency of 20% of the total planned samples.

7.0 Analytical Methods

Samples collected during the Building 3 sampling effort will be analyzed by the subcontractor laboratory. This laboratory must be certified by the USACE Center of Expertise. QA samples shall be collected and analyzed by the designated USACE QA Laboratory.

The subcontractor laboratory supporting this work shall provide statements of qualifications including organizational structure, QA Manual, and standard operating procedures (SOPs). Laboratory standard operating procedures are based on the methods as published by the EPA in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW846*, Third/Fourth Edition (November 1986; Revision 1, July 1992; Revision 2, November 1992; and Updates 1, 2, and 3). These SOPs must be adapted from and reference standard EPA SW-846 methods and thereby specify:

- Procedures for sample preparation
- Instrument start-up and performance check
- Procedures to establish the actual and required detection limits for each parameter
- Initial and continuing calibration check requirements
- Specific methods for each sample matrix type
- Required analyses and QC requirements

Samples collected during the project will be analyzed by EPA SW-846 methods. The analytes of interest and the corresponding SW-846 methods to be used for this project are presented in Table 3-1. The primary SW-846 methods include:

- Method 8082 PCBs
- Method 8270C SVOCs
- Method 6010B Metals (except mercury)
- Method 7470A/74741A Mercury
- Method 8015B Modified (M) Petroleum Hydrocarbons (GRO/DRO)

Tables 7-1 through 7-8 present the reporting limits and precision and accuracy limits for each of the primary analytical method. The subcontract laboratory shall submit SOPs detailing the specific MDLs for each analytical method.

If contaminant concentrations are high, or if matrices (other than normal waters and soils) create a problematic effect on the analysis, analytical protocols may require modifications to defined

methodology. Any proposed changes to standard analytical methods require written approval from the Contractor and CENWK. All analytical method variations will be identified in project addenda. These may be submitted for regulatory review and approval when directed by the CENWK Project Manager.

7.1 Preparation Procedures

Extraction and digestion procedures for the preparation of solid and liquid matrices will include the following:

- Method 1311 Toxicity Characteristic Leaching Procedure (TCLP): Method 1311 is used to prepare samples for the determination of the concentration of organic and inorganic constituents that are leachable from waste or other material.
- Method 3005A Acid Digestion of Water Samples for Metals Analysis: Method 3005A consists of an acid digestion procedure to prepare aqueous samples for metals analysis. The digested samples are analyzed for total recoverable and dissolved metals determination by inductively couple plasma spectroscopy (ICP).
- Method 3010A Acid Digestion of Aqueous Samples and Extracts for Metals Analysis: Method 3010A prepares aqueous or waste samples for total metals determination by ICP.
- Method 3540 or 3541 Soxhlet Extraction: Methods 3540 and 3541 are procedures for extracting nonvolatile and semivolatile organic compounds from solids such as soils and sludges. Method 3541 is an automated Soxhlet extraction. The soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent. [Note: For the Building 3 project, the laboratory has the option of performing either Method 3540 or 3541 for sample extraction for PCB analysis.]
- Method 3580A Waste Dilution: This method involves a solvent dilution of a non-aqueous waste sample prior to analysis. This method is used in combination with Method 1311 for preparing samples for TCLP analysis.

7.2 Analytical Procedures

Analytical methods for solid and water matrices associated with this project will include:

• Method 8082 - Polychlorinated Biphenyls (PCBs) by Gas Chromatography: Method 8082 is used to determine the concentrations of PCBs as Aroclors or as individual PCB congeners in extracts from solid and aqueous matrices. Open-tubular, capillary columns are employed with electron capture detectors (ECD) or electrolytic conductivity detectors (ELCD).

- Method 8270C Semivolatile Organic Compounds (SVOCs): Semivolatile organic compounds (also known as base-neutral and acid extractables) in water and soil samples are analyzed using method Method 8270C. This technique quantitatively determines the concentration of a number of SVOCs. Samples are solvent extracted and concentrated through evaporation of the solvent. Compounds of interest are separated and quantified using a capillary column GC/mass spectrometer.
- Method 6010B Trace Metals by Inductively Coupled Plasma Atomic Emission Spectroscopy for Water and Soils: Samples are analyzed for trace metals using Method 6010B for water and soils. Analysis for most metals requires digestion of the sample. Following digestion, the trace elements are determined simultaneously or sequentially using Inductively Coupled Plasma Atomic Emission Spectroscopy (ICPAES).
- **Method 7470A/7471A Mercury Manual Cold-Vapor Technique**: Water and soil samples are analyzed for mercury using methods SW7470A and SW7471A, respectively. This method is a cold-vapor, flameless atomic absorption (AA) technique based on the absorption of radiation by mercury vapor.
- Method 8015B(M) Nonhalogenated Organics Using GC/FID: Method 8015B is used to determine the concentration of various nonhalogenated volatile organic compounds and semivolatile organic compounds by gas chromatography. Method 8015B is applicable to the analysis of petroleum hydrocarbons, including gasoline range organics (GROs) and diesel range organics (DROs).

8.0 Laboratory Calibration Procedures

This section describes procedures for maintaining the accuracy of all the instruments and measuring equipment that are used for conducting laboratory analyses. These instruments and equipment shall be calibrated before each use or on a scheduled, periodic basis according to manufacturer instructions.

8.1 Analytical Support Areas

The following sections discuss the calibration needs for operations within the analytical laboratory necessary to support the instrumentation.

8.1.1 Analytical Standards

All primary reference and secondary working standards used for the purpose of instrument calibration and recovery determinations must be traceable to National Institute of Standards and Technology (NIST) or EPA sources. The preparation and use of these standards must be documented in a standards log book which shall include the preparers name, date of preparation, and date of expiration and storage location.

8.1.2 Laboratory Balances

All balances to be used for sample weights and/or standards preparation must receive an annual manufacturer's calibration. The balance must be calibrated daily with a minimum of two class "S" weights which bracket the range of weights to be determined. A hardbound balance logbook must be maintained with the results of the daily calibrations.

8.1.3 Laboratory Refrigerators/Freezers

All cold storage units (for both samples and standards) must be monitored daily for proper use. The acceptable working range of the unit must be clearly posted on the unit's front panel. All thermometers used for monitoring must be immersion type and be calibrated versus a certified thermometer on a yearly basis.

8.1.4 Laboratory Water Supply

The laboratory water unit shall be capable of supplying sufficient quantities of American Society for Testing and Materials (ASTM) Type II reagent water (resistivity of >1 megohm-cm @25 C) to the required analytical areas. Recommendations for "polishing" water for analytical use are ion-exchange units for inorganic analyses and distillation/deionization followed by UV treatment

or carbon absorption for organic analyses. Conductivity or resistance reading of the system water shall be documented minimally daily or greater dependant upon the water usage.

8.2 Laboratory Analytical Instrumentation

Details regarding the procedures for calibration of laboratory equipment and maintenance of calibration records will be presented in laboratory QA Plans and/or SOPs. These procedures will be reviewed by the Contractor and USACE prior to the start of sampling and analysis activities. For all analyses conducted according to SW-846, the calibration procedures and frequencies specified in the SW-846 methods will be followed. Tables 8-1 through 8-4 present a summary of the standard calibration procedures for the project-specific analytical methods, except Method 8015B(M). Although these tables are provided for general reference, the subcontract laboratory shall submit SOPs and/or QA Plants detailing the specific calibration procedures (including acceptance criteria and corrective actions) to be used for each analytical method.

Records of calibration will be kept as follows:

- Each instrument will have a record of calibration with an assigned record number.
- A label will be affixed to each instrument showing identification numbers, manufacturer, model numbers, date of last calibration, signature of calibrating analyst, and due date of next calibration. Reports and compensation or correction figures will be maintained with instrument.
- A written step-wise calibration procedure will be available for each piece of test and measurement equipment.
- Any instrument that is not calibrated to the manufacturer's original specification will
 display a warning tag to alert the analyst that the device carries only a "Limited
 Calibration."

Records of calibration, repairs, or replacement will be filed and maintained by laboratory personnel performing QC activities. These records will be filed at the location where the work is performed and will be subject to QA audit.

9.0 Laboratory QA/QC

The subcontractor laboratory will have a written QA program that provides guidelines to ensure the reliability and validity of work conducted at the laboratory. The objectives of the laboratory QA program will be to:

- Properly collect, preserve, and store all samples
- Maintain adequate custody records from sample collection through reporting and archiving of results
- Use properly trained analysts to analyze all samples by approved methods within holding times
- Produce defensible data with associated documentation to show that each system was calibrated and operating within precision and accuracy control limits
- Accurately calculate, check, report, and archive all data using the Laboratory Information Management System (LIMS)
- Document all the above activities so that project data can be independently validated.

Laboratory QA Plans will be appropriately referenced and implemented in their entirety. Compliance with the QA program will be coordinated and monitored by the laboratory's QA department, which is independent of the operating departments.

To ensure the production of analytical data of known and documented quality, the subcontractor laboratory will implement method and batch QC checks as described below. Internal quality control checks are generated by the analytical laboratory and are used to determine whether an analytical operation is in control or if the sample matrix has an effect on the data being generated. Internal QC measures for analysis will be conducted in accordance with SOPs and the individual method requirements. The minimum QC requirements for SW-846 methods (excluding 8015B) proposed for use at Building 3 are presented in Tables 8-1 through 8-4, including the types of QC checks, the frequency for implementation of each QC measure, and the acceptance criteria for the QC check. Although these tables are provided for general reference, the subcontract laboratory shall submit SOPs and/or QA Plants detailing the specific calibration procedures (including acceptance criteria and corrective actions) to be used for each analytical method.

The laboratory will provide documentation in each data package that both initial and ongoing instrument and analytical QC functions have been met. Any non-conforming analyses will be reanalyzed by the laboratory, if sufficient sample volume is available. It is expected that sufficient sample volumes will be collected to provide for re-analyses, if required. Tables 8-1

through 8-4 present general QC acceptance criteria and corrective actions for the applicable analytical methods. However, the specific QC protocols used by the subcontractor laboratory will be documented in the Laboratory QA Plan and/or SOPs.

9.1 Batch Quality Control

Sample batch QC can either be associated with sample preparation or with the analytical determination. In either case, the batch is not to exceed twenty samples of similar matrix. The *preparation batch* is the set of samples, which are extracted or digested together by the same lab technician, with the same lot of reagents, over the same time. All the samples within the same preparation batch must be of the same matrix, and the batch must have its own unique method blank and QC samples as defined below. The *analytical batch* is the group of samples that are analyzed together during the same analytical sequence within one continuous time period. The analytical batch can consist of multiple preparation batches but must analyze all constituents of the preparation batch. QC cannot be run separate from the analytical samples.

9.1.1 Method Blanks

There are two types of method blanks –instrument blanks and preparation blanks. An instrument blank is an aliquot of pure, non-contaminated reagent (i.e. reagent water) that is analyzed prior to samples to establish background levels of the analytical system. The preparation blank is a sample of a pure, non-contaminated matrix of interest (usually reagent grade water or purified silica sand) that is subjected to all of the sample preparation (digestion, distillation, extraction) and analytical methodology applied to the samples. The preparation blank is used to assess the level of background contamination which might affect low level concentration results. The affect could be either false positive results or biased high concentration results. Method blanks must be prepared and analyzed with each analytical sample batch. Method blanks will be evaluated against MDLs in accordance with CLP National Functional Guidelines. Contamination levels reported in the blanks are never subtracted from the concentration of the sample.

9.1.2 Laboratory Control Samples (LCS)

The LCS contains known concentrations of analytes representative of the contaminants to be determined and is carried through the entire preparation and analysis process. The primary purpose of the LCS is to establish and monitor the laboratory's analytical performance control. Commercially available LCSs or those from EPA may be used. LCS standards prepared in-house must be made from a source independent of that of the calibration standards. An LCS must be

analyzed with each analytical sample batch. The results (as percent recovery) for each LCS analyte must be plotted on a control chart.

9.1.3 Laboratory Duplicates

Laboratory duplicates are separate sample weights of a single sample that are prepared and analyzed concurrently at the laboratory. This duplicate sample shall not be a method blank, trip blank, or field blank. The primary purpose of the laboratory duplicate is to check the precision of the laboratory analyst, the sample preparation methodology, and the analytical methodology. If there are significant differences between the duplicates, the affected analytical results will be re-examined. One in 20 samples will be a laboratory duplicate, with fractions rounded to the next whole number.

9.1.4 Surrogate Spikes

A surrogate spike is prepared by adding a pure compound to a sample before extraction. The compound in the surrogate spike should be of a similar type to that being assayed in the sample. The purpose of a surrogate spike is to determine the efficiency of recovery of analytes in the sample preparation and analysis. The percent of recovery of the surrogate spike is then used to gauge the total accuracy of the analytical method for that sample. The frequency for performing surrogate spikes is dependent on the analytical method.

9.1.5 Matrix Spikes and Matrix Spike Duplicates

An MS is a second sample weight of the original sample spiked with known quantities of analytes and subjected to the entire analytical procedure. It is used to indicate the appropriateness of the method for the matrix by measuring recovery. An MSD is a second sample weight of the same sample with known quantities of compounds added. The purpose of the MSD is to evaluate method precision. MSs and MSDs are performed at a frequency of one per 20 samples of similar matrix.

Note: For this project, MS/MSDs will be analyzed using sites-specific samples. For analysis of PCBs, the spiking concentration shall be 50 ppm.

9.2 Method-Specific Quality Control

The laboratory must follow specific quality processes as defined by the analytical method. These will include measures such as calibration verification samples, instrument blank analysis, internal

standards implementation, method of standard additions utilization, serial dilution analysis, post-digestion spike analysis, etc.

10.0 Laboratory Preventative Maintenance

As part of the laboratory's QA/QC program, a routine preventive maintenance program will be implemented to minimize the occurrence of instrument failure and other system malfunctions. All laboratory instruments will be maintained in accordance with manufacturers' specifications and the requirements of the specific method employed. This maintenance will be carried out on a regular, scheduled basis and will be documented in the laboratory instrument service log book for each instrument. Emergency repair or scheduled manufacturer's maintenance will be provided under a repair and maintenance contract with factory representatives. Table 10-1 of this QAPP provides typical maintenance items for select equipment associated with this project; however; this table is not intended to be inclusive of all required preventative maintenance procedures. The subcontractor laboratory shall provide written preventative maintenance in the laboratory-specific QA Plan and/or SOPs.

11.0 Analytical Corrective Actions

Corrective actions may be required for two major types of problems: analytical/equipment problems and noncompliance with acceptance criteria. Analytical and equipment problems may occur during sampling, sample handling, sample preparation, laboratory instrumental analysis, and data review.

The laboratory-specific QA Plan shall provide systematic procedures to identify laboratory related out-of-control situations and corrective actions. Corrective actions shall be implemented to resolve problems and restore malfunctioning analytical systems. Laboratory personnel will have received QA training and will be aware that corrective actions are necessary when:

- QC data are outside warning or control windows for precision and accuracy
- Blanks contain target analytes above acceptable levels and must be investigated
- Undesirable trends are detected in spike recoveries or RPD between duplicates
- There are unusual changes in detection limits
- Deficiencies are detected by internal audits, external audits, or from performance evaluation samples results
- Inquiries concerning data quality are received.

Corrective action procedures are often handled at the bench level by the analyst who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, prepares spike and calibration mixes, checks instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the Laboratory Supervisor, Manager, and/or QA Department for further investigation. Once resolved, full documentation of the corrective action procedure is filed with project records and the QA Department, and the information is summarized within case narratives.

Typical analytical corrective actions include:

- Re-analyzing the samples, if holding time criteria permit
- Re-extraction and re-analysis, if holding time criteria permit
- Evaluating blank contaminant sources, elimination of these sources, and reanalysis
- Modifying the analytical method (i.e., standard additions) with appropriate notification and documentation
- Re-sampling and analyzing
- Evaluating and amending sampling procedures

Accepting data and acknowledging the level of uncertainty.

If re-sampling is deemed necessary due to laboratory problems, the Contractor and CENWK Project Manager will evaluate the costs/benefits of implementing the additional sampling effort.

11.1 Incoming Samples

Problems noted during sample receipt will be documented in the appropriate laboratory letter-of-receipt (LOR). The Contractor and CENWK Project Manager will be contacted immediately to determine resolution to the problem. All corrective actions will be thoroughly documented.

11.2 Sample Holding Times

When sample extraction/digestion or analytical analyses are not performed within method required holding times, the Contractor and CENWK Project Chemist will be notified immediately to determine resolution to the problem. Resampling is the most probable corrective action for expired holding time. If holding times are exceeded due to laboratory oversight, resampling will be conducted at laboratory's expense. All corrective actions will be thoroughly documented.

11.3 Instrument Calibration

Project samples shall not be analyzed by instrumentation which fails to meet tuning and/or standardization/calibration criteria as presented laboratory SOPs and/or QA Plans (referencing Tables 8-1 through 8-4). All project samples will be reanalyzed if performed following an initial and/or continuing calibration analytical sequence that does not meet method requirements. Corrective action may require standard re-preparation, instrument maintenance, and instrument recalibration /restandardization.

11.4 Reporting Limits

All appropriate measures shall be required to prepare samples in an attempt to achieve the reporting limits as stated in Tables 7-1, 7-3, 7-5, 7-7 and 7-8. When difficulties arise in achieving these limits, the laboratory will notify the Contractor and CENWK Project Chemist to determine problem resolution. All corrective actions shall be thoroughly documented.

Any dilutions impacting the reporting limits will be documented in case narratives along with revised reporting limits for those analytes affected. Analytes detected above the method

detection limits, but below the reporting limits, will be reported as estimated values. Both the undiluted and diluted set of data shall be provided to the Contractor.

11.5 Method Quality Control

Failure of method-required QC to meet the requirements specified in laboratory SOPs/QA Plans (referencing Tables 8-1 through 8-4 of this QAPP) shall require corrective actions for all affected data. The Contractor and CENWK Project Chemist will be notified as soon as possible to discuss possible corrective actions, particularly when unusual or difficult sample matrices are encountered.

11.6 Calculation Errors

When calculation or reporting errors are noted within any given data package, reports will be reissued with applicable corrections. Case narratives will clearly state the reasons for reissuance of reports.

12.0 Calculation of Data Quality Indicators

Laboratory results will be assessed for compliance with required precision, accuracy, completeness, sensitivity and representativeness/comparability as outlined in the following sections.

12.1 Precision

The precision of the laboratory analytical process will be determined through evaluation of the comparative determination of the LCS and LCSD, the MS and MSD, and/or the sample and sample duplicate analyses. Investigative sample matrix precision will be assessed by comparing the analytical results between MS/MSD for organic analysis and laboratory duplicate analyses for inorganic analysis. (MS/MSD pairs may also be prepared for inorganic analyses). The RPD will be calculated for each pair of duplicate analysis using appropriate formulas in Table 12-1 and produce an absolute value for RPD. This precision measurement will include variables associated with the analytical process, influences related to sample matrix interferences, and sample heterogeneity.

12.2 Accuracy

The accuracy of the laboratory analytical measurement process will be determined by comparing the percent recovery for the LCS / LCSD versus its documented true value. Overall project accuracy includes the assessment of investigative sample using the analytical results of MS and MSD samples. The percent recovery (%R) of LCS and MS/MSD samples will be calculated using the appropriate formula in Table 12-1. This overall accuracy will include variables associated with the analytical process, influences related to sample matrix interferences, and sample heterogeneity.

12.3 Data Completeness

Data completeness of laboratory analyses will be assessed for compliance with the amount of data required for decision making. The completeness is calculated using the following equation:

Completeness objectives were defined in Section 3.3.

12.4 Project Completeness

Project completeness will be determined by evaluating the planned versus actual data. Consideration will be given for project changes and alterations during implementation. All data not flagged as rejected by the review, verification, validation, or assessment processes will be considered valid. Overall, the project completeness will be assessed relative to media, analyte, and area of investigation. Completeness objectives were defined in Section 3.3.

12.5 Sensitivity

Sensitivity of the analytical determination is directly related to the laboratory's MDL. Achieving MDLs depends on sample preparation techniques, instrumental sensitivity, and matrix effects. Therefore, it is important to determine actual MDL through the procedures outlined in 40 CFR 136, Appendix C. MDLs should be established for each major matrix under investigation (i.e., concrete, soil, water) through multiple determinations, leading to a statistical evaluation of the MDL.

It is important to monitor instrument sensitivity through calibration blanks and low concentration standards to ensure consistent instrument performance. It is also critical to monitor the analytical method sensitivity through analysis of method blanks, calibration check samples, and LCSs, etc.

12.6 Representativeness/Comparability

Representativeness expresses the degree to which data accurately reflect the analyte or parameter of interest for the environmental media examined at the site. It is a qualitative term most concerned with the proper design of the sampling program. Factors that affect the representativeness of analytical data include appropriate sample population definitions, proper sample collection and preservation techniques, analytical holding times, use of standard analytical methods, and determination of matrix or analyte interferences. Sample collection, preservation, analytical holding time, analytical method application, and matrix interferences will be evaluated by reviewing project documentation and QC analyses.

Comparability, like representativeness, is a qualitative term relative to the confidence of how one project data set compares with another. The comparability issue is controlled through the use of defined sampling methodologies, use of standard sampling devices, standard analytical protocols/procedures, and QC checks with standard control limits. Through proper implementation and documentation of these standard practices, the project will establish confidence that data will be comparable to other project and programmatic information.

Additional input to determine representativeness and comparability may be gained through statistical evaluation of data populations, chemical charge balances, compound evaluations, or dual measurement comparisons.

13.0 Data Validation, Reduction, and Reporting

This chapter describes the data review process enacted to ensure validity of the analytical data. All data generated by the analytical laboratory will be initially reviewed by the laboratory technical personnel prior to being submitted to the Contractor. This review will provide a check to ensure the correctness of the reported results and generate a case narrative to explain any anomalies which may affect the validity or useability of the data. Following receipt of the data package, the electronic data will be validated by the database and the hardcopy data will be validated by the Contractor chemists or designees.

13.1 Data Reduction

Data reduction requirements apply to both field data and laboratory-generated data.

13.1.1 Field Data

Raw data from field measurements and sample collection activities will be appropriately recorded in field logbooks. Data to be used in project reports will be reduced and summarized. The methods of data reduction will be documented.

The Contractor Project Manager or designee is responsible for data review of all field-generated data. This includes verifying that all field descriptive data are recorded properly, that all field instrument calibration requirements have been met, that all field QC data have met frequency and criteria goals, and that field data are entered accurately in all logbooks and worksheets.

13.1.2 Laboratory Data

All samples collected for the project will be sent to a USACE-approved laboratory. Data reduction, evaluation, and reporting of samples analyzed by the laboratory will be performed according to specifications outlined in both the laboratory's QA Plans and this QAPP. Laboratory reports will include documentation verifying analytical holding time compliance.

The laboratory will perform in-house analytical data reduction under the direction of the Laboratory QA Manager. The Laboratory QA Manager or designee are ultimately responsible for assessing data quality and informing the Contractor and CENWK of any data which are considered "unacceptable" or require caution on the part of the data user in terms of its reliability. Data will be reduced, reviewed, and reported as described in the laboratory QA Plans.

Data reduction, review, and reporting activities performed by the laboratory are summarized below:

- Raw data are produced by the analyst who has primary responsibility for the accuracy and completeness of the data. All data will be generated and reduced following the QAPP defined methods and implementing laboratory SOP protocols.
- Level 1 technical data review is completed relative to an established set of guidelines by a peer analyst. The review shall ensure the completeness and correctness of the data while assuring all method QC measures have been implemented and were within appropriate criteria. Items to be reviewed include: preparation logs, analysis runs, methodology, results quality control results, internal QC checks, checklists and sign off sheets.
- Level 2 technical review is completed by the area supervisor or data review specialist. This reviews the data for attainment of QC criteria as outlined in the established methods and for overall reasonableness. It will ensure all calibration and QC data are in compliance, qualitative identification of compounds is correct, quantitative calculations are correct, and check at least 10 percent of the data calculations. This review shall document that the data package is complete and ready for reporting and archival.
- Upon acceptance of the raw data by the area supervisor, the report is generated and sent to the Laboratory Project Manager or QA representative for Level 3 administrative data review. This total overview review will ensure consistency and compliance with all laboratory instructions, the laboratory QA Plans, the project laboratory SOW, and the project QAPP.
- The Laboraory Project Manager will complete a thorough review of all reports.
- Final reports will be generated and signed by the Laboratory Project Manager.
- Data packages, in Contract Laboratory Program (CLP) format, will then be delivered to the Contractor for data validation (refer to Table 13-1).

The data review process will include identification of any out-of-control data points and data omissions, as well as interactions with the laboratory to correct data deficiencies. Decisions to repeat sample collection and analyses may be made by the Project Manager based on the extent of the deficiencies and their importance in the overall context of the project. The laboratory will provide flagged data to include such items as:

- Concentration below required detection limit
- Estimated concentration due to poor spike recovery
- Concentration of chemical also found in laboratory blank

13.2 Data Validation

Data validation is the systematic review process performed to ensure that the precision and accuracy of the analytical data are adequate for their intended use.

13.2.1 Data Validation Approach

The greatest uncertainty in a measurement is often a result of the sampling process and inherent variability in the environmental media rather than the analytical measurement. Therefore, analytical data validation will be performed only to the level necessary to minimize the potential of using false positive or false negative results in the decision-making process (i.e., to ensure accurate identification of detected versus non-detected compounds). This approach is consistent with the DQOs for the project, with the analytical methods, and for determining contaminants of concern and calculating risk.

Samples will be analyzed through use of standard analytical methods. Definitive data will be reported consistent with the deliverables identified in Section 13.1.2 and Table 13-1. This report content is consistent with what is understood as an EPA Level IV deliverable (data forms including laboratory QC, and raw sample data including calibration information). Definitive data will then be validated through the review process presented in Section 13.2.2 and qualified using guidelines established by the analytical method. DQOs identified in Section 3.0 and method-specified criteria will be validated. An additional copy of the comprehensive analytical information will be retained by the subcontract laboratory.

13.2.2 Primary Data Validation Categories

Validation will be performed by comparing the contents of the complete data package (raw data, sample results and QA/QC results) to the requirements established both in the requested analytical methods and the criteria presented in this QAPP. The Contractor Validation support staff will be responsible for these activities. The protocols for analytical data validation are presented in:

- SW-846 Analytical Method Requirements
- EPA CLP National Functional Guidelines for Organic Data Review (EPA 1994b)
- EPA CLP National Functional Guidelines for Inorganic Data Review (EPA 1994c)

The data will be validated using the processes and procedures provided in the National Functional Guidelines, but the guidelines used for control will be the criteria established and presented within the SW-846 methods.

- Holding Times Evaluation of holding times ascertains the validity of results based on the length of time from sample collection to sample preparation or sample analysis. Verification of sample preservation must be confirmed and accounted for in the evaluation of sample holding times. The evaluation of holding times is essential to establishing sample integrity and representativeness. Concerns regarding physical, chemical, or biochemical alteration of analyte concentrations can be eliminated or qualified through this evaluation.
- Blanks The assessment of blank analyses is performed to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks applies to any blank associated with the samples, including field, trip, equipment, and method blanks. Contamination during sampling or analysis, if not discovered, results in false-positive data. Blanks will be evaluated against MDLs in accordance with CLP National Functional Guidelines. Field, trip, and equipment rinsate blanks will be evaluated against 5× MDLs for most analytes and 10× the MDLs for common laboratory solvent analytes.
- Laboratory Control Samples The LCS serves as a monitor of the overall performance of the analytical process, including sample preparation, for a given set of samples. Evaluation of this standard provides confidence in or allows qualification of results based on a measurement of process control during each sample analysis.
- Surrogate Recovery System monitoring compounds are added to every sample, blank, matrix spike, MS, MSD, and standard. They are used to evaluate extraction, cleanup, and analytical efficiency by measuring recovery on a sample-specific basis. Poor system performance as indicated by low surrogate recoveries is one of the most common reasons for data qualification. Evaluation of surrogate recovery is critical to the provision of reliable sample-specific analytical results.
- Internal Standards Internal standards are utilized to evaluate and compensate for sample-specific influences on the analyte quantification. They are evaluated to determine if data require qualification due to excessive variation in acceptable internal standard quantitative or qualitative performance measures. For example, a decrease or increase in internal standard area counts for organic compounds may reflect a change in sensitivity that can be attributed to the sample matrix. Because quantitative determination of analytes is based on the use of internal standards, evaluation is critical to the provision of reliable analytical results.

- Furnace Atomic Absorption Quality Control Duplicate injections and furnace postdigestion spikes are evaluated to establish precision and accuracy of individual analytical determinations. Because of the nature of the furnace atomic absorption technique and because of the detailed decision tree and analysis scheme required for quantitation of the elements, evaluation of the QC is critical to ensuring reliable analytical results.
- Calibration The purpose of initial and continuing calibration verification analyses is to verify the linear dynamic range and stability of instrument response. Relative instrument response is used to quantitate the analyte results. If the relative response factor is outside acceptable limits, the data quantification is uncertain and requires appropriate qualification.
- Sample Reanalysis When instrument performance-monitoring standards indicate an analysis is out of control, the laboratory is required to reanalyze the initial sample. If the analysis is out of control again, the sample must then be re-prepared and analyzed. If the reanalyses do not solve the problem (i.e., surrogate compound recoveries are outside the limits for both analyses), the laboratory is required to submit data from both analyses. An independent review is required to determine which is the appropriate sample result.
- Secondary Dilutions When the concentration of any analyte in any sample exceeds the initial calibration range, a new aliquot of that sample must be diluted and reanalyzed. The laboratory is required to report data from both analyses. When this occurs, an independent review of the data is required to determine the appropriate results to be used for that sample. An evaluation of each analyte exceeding the calibration range must be made, including a review of the dilution analysis performed. Results chosen in this situation may be a combination of both the original results (i.e., analytes within initial calibration range) and the secondary dilution results.
- Raw Data (inc. Chromatograms and Intensity/Absorbance Readings) Raw data will be used to assess the qualitative and quantitative assumptions and decisions made by the laboratory and determine whether the decisions made within the laboratory are substantiatible from a third party position. Retention times and identifications of tentatively identified compounds are verified.
- Laboratory Case Narratives Analytical laboratory case narratives are reviewed for specific information concerning the analytical process. This information is used to direct the data validator to potential problems with the data.

13.3 Data Reporting

The analytical laboratory will provide all project data in both hardcopy and electronic format as discussed below. The laboratory will also be required to confirm sample receipt and log-in information. The laboratory will return a copy of the completed COC and confirmation of the laboratory's analytical log-in to the Contractor within 24 hours of sample receipt.

For all (100%) project data, the subcontract analytical laboratory will prepare and deliver a full copy of an analytical data package as required for CLP Level III. At a minimum, the following information will be provided in each analytical data package submitted:

- Cover sheets listing the samples included in the report and narrative comments describing problems encountered in analysis
- Tabulated results of inorganic, organic, and other parameters identified and quantified
- Analytical results for QC sample spikes, sample duplicates, initial and continuous calibration verifications of standards and blanks, standard procedural blanks, LCSs, etc.

Additionally, upon review of the Level III data deliverables, the Contractor will randomly select 10% of the data packages for Level IV validation as described below. At the request of the Contractor, the analytical laboratory will provide a CLP Level IV data package for the specified results. The Level IV package will include all Level III information in addition to the following:

- Associated raw data to support the tabulated results for samples and QA/QC
- Tabulation of instrument detection limits determined in pure water.

The lab is required to retain a full copy of the analytical and QC documentation. Such retained documentation will include all hard copies and electronic storage media (e.g., magnetic tape). As needed, the analytical laboratory will supply hard or electronic copies of the retained information.

The data are required to be formatted in a database format, as specified by the Contractor, to facilitate electronic data entry, review, and evaluation. The electronic data set will be transferred automatically into the project database. Following the transfer, the data set will be validated to an equivalent EPA Level III validation review. As part of the review, an error report will be generated from the database, which includes data flags in accordance with the above-referenced protocols. The report will be accompanied with additional comments of the data validator(s). The associated data flags will include such items as: (1) estimated concentration below-required reporting limit; (2) estimated concentration due to poor calibration, internal standard, or surrogate recoveries; (3) estimated concentration due to poor spike recovery; and (4) estimated

concentration of chemical that was also determined in the laboratory blank. The EPA Level III validation review will apply to 100% of project data.

After the electronic validation has been performed, an EPA Level IV validation on a minimum of 10% of the data will be performed by qualified chemists. Flags signifying the usability of data will be noted and entered into an analytical data base. Deficiencies in data deliverables will be corrected through direct communication with the laboratory, generating immediate response and resolution. All significant data discrepancies noted during the validation process will be documented through NCRs, which are sent to the laboratory for clarification and correction. Decisions to repeat sample collection and analyses may be made by the Contractor Project Manager and the Project Chemist based on the extent of the deficiencies and their importance in the overall context of the project.

Data assessment will be accomplished by the joint efforts of the data validator, the Project Chemist and the Project Manager. Data assessment will be based on the criteria that the sample was properly collected and handled according to the FSP and QAPP. An evaluation of data accuracy, precision, sensitivity and completeness, based on criteria presented in this QAPP, will be performed by the data validator and presented in the QCSR. This data quality assessment will indicate that data are: (1) usable as a quantitative concentration, (2) usable with caution as an estimated concentration, or (3) unusable due to excessive out-of-control QC results.

Project data sets will be available for controlled access by the Contractor Database Manager and other authorized personnel. Each data set will be incorporated into project reports as required.

13.4 Data Turnaround Time Requirements

The turnaround time for analytical deliverables for the Building 3 sampling effort is 7 days, although the Contractor is not restricted from requiring accelerated turnaround times upon request. Sufficient notification time will be provided by the Contractor prior to decreasing the turnaround time.

14.0 Performance and System Audits

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the FSP and QAPP. Audits of laboratory activities will include both internal and external audits.

14.1 External Laboratory Audits

The USACE HTRW CX conducts on-site audits and validates laboratories on a regular basis. These USACE independent on-site systems audits in conjunction with performance evaluation samples (performance audits) qualify laboratories to perform USACE environmental analysis every 18 months.

These system audits include examining laboratory documentation of sample receiving, sample log-in, sample storage, COC procedures, sample preparation and analysis, instrument operating records, etc. Performance audits consist of sending performance evaluation samples to USACE laboratories for on-going assessment of laboratory precision and accuracy. The analytical results of the analysis of performance evaluation samples are evaluated by USACE HTRW CX to ensure that laboratories maintain an acceptable performance.

14.2 Internal Laboratory Audits

Internal performance and system audits of laboratories will be conducted by the Laboratory QA Officer as directed in the laboratory QA Plans. These system audits will include examination of laboratory documentation of sample receiving, sample log-in, sample storage, COC procedures, sample preparation and analysis, instrument operating records, etc. Internal performance audits are also conducted on a regular basis. Single-blind performance samples are prepared and submitted along with project samples to the laboratory for analysis. The Laboratory QA Officer will evaluate the analytical results of these single-blind performance samples to ensure that the laboratory maintains acceptable performance.

15.0 QA Reports and Documentation

This section describes the primary quality assurance reports to be prepared by the Contractor and submitted to USACE project management.

15.1 Daily Chemical Data Reports

During field activities, the Contractor will prepare Daily Quality Control Reports (DQCRs) as described in the FSP. In addition to the item specified in the FSP, a daily analytical data report will be included as an attachment to the DQCR. This report will present tabulated analytical results for data that was received since the prior DQCR was submitted to USACE.

15.2 Laboratory Quality Assurance Reports

Each laboratory will provide LORs and analytical QC summary statements (case narratives) with each data package. All COC forms will be compared with samples received by the laboratory and a LOR will be prepared and sent to the Contractor describing any differences in the COC forms and the sample labels or tags. All deviations will be identified on the receiving report such as broken or otherwise damaged containers. This report will be forwarded to the Contractor within 24 hours of sample receipt and will include the following: a signed copy of the COC form; itemized sample numbers; laboratory sample numbers; cooler temperature upon receipt; and itemization of analyses to be performed. Summary QC statements will accompany analytical results as they are reported by the laboratory in the form of case narratives for each sample delivery group.

15.3 Quality Control Summary Reports

At the conclusion of field investigation activities and laboratory analysis, the Contractor, in addition to any review conducted by the laboratory, will perform its own validation of the submitted data. This activity will include assignment of flags to data, documentation of the reason(s) for the assignments, and description of any other data discrepancies. The Contractor will then prepare a Quality Control Summary Report (QCSR), which will be included as an appendix to the final report. This report will be submitted to the CENWK Project Manager as determined by the project schedule. The contents of the QCSR will include data validation documentation and discussion of all data that may have been compromised or influenced by aberrations in the sampling and analytical processes. Both field and laboratory QC activities will be summarized, and all DQCR information will be consolidated. Problems encountered, corrective actions taken, and their impact on project DQOs will be determined.

The following are examples of elements to be included in the QCSR, as appropriate:

- Laboratory QC evaluation and summary of the data quality for each analytical type and matrix. Part of the accuracy, precision, and sensitivity summarized in the data quality assessment.
- Field QC evaluation and summary of data quality relative to data useability. Part of the accuracy, precision, and sensitivity summarized in the data quality assessment.
- Overall data assessment and usability evaluation.
- DCQCR consolidation and summary.
- Summary of lessons learned during project implementation.

Specific elements to be evaluated within the QCSR include the following:

- Sample results
- Field and laboratory blank results
- Laboratory control sample percent recovery (method dependent)
- Sample matrix spike percent recovery (method dependent)
- Matrix spike/matrix spike duplicate or sample duplicate RPD (method dependent)
- Analytical holding times
- Surrogate recovery, when appropriate.

15.4 Field Work Variances

Any departures from approved plans will receive prior approval from the CENWK Project Manager and will be documented via Field Work Variances (FWVs) as discussed in Section 9.3 of the FSP. FWVs will be incorporated into the project evidence file.

15.5 Project Evidence Files

The Contractor will maintain custody of the project evidence file and will maintain the contents of files for this project, including all relevant records, reports, logs, field logbooks, pictures, subcontractor reports, correspondence, and COC forms, until this information is transferred to the CENWK Project Manager. These files will be stored under custody of the Contractor Project Manager. The analytical laboratory will retain all original analytical raw data information (both hard copy and electronic) in a secure, limited access area and under custody of the laboratory Project Manager.

16.0 References

- ASTM (American Society of Testing and Materials). 1996. <u>Annual Book of ASTM Standards</u>, Volume 04.08, Soil and Rock.
- EPA (U. S. Environmental Protection Agency) 1985. <u>NEIC Policies and Procedures</u>, EPA-300/9-78DDI-R, Revised June.
- EPA 1991. <u>Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans</u>, QA/R5, revised October
- EPA 1993a. Data Quality Objectives Process, EPA-540-R-93-071, September.
- EPA 1993b. <u>Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition</u>, Revision 1, Update 1.
- EPA 1994a. EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, EPA QA/R-5, January.
- EPA 1994b. <u>EPA Contract Laboratory Program National Functional Guidelines for Organic</u>
 Data Review, EPA-540/R-94/012, February.
- EPA 1994c. <u>EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review</u>, EPA-540/R-94/013, February.
- USACE (U. S. Army Corps of Engineers) 1994. <u>Requirements for the Preparation of Sampling and Analysis Plans</u>, EM 200-1-3, September.
- USACE (U. S. Army Corps of Engineers) 1998. <u>Chemical Data Quality Management for Hazardous, Toxic, Radioactive Waste Remedial Activities</u>, ER 1110-1-263, April.

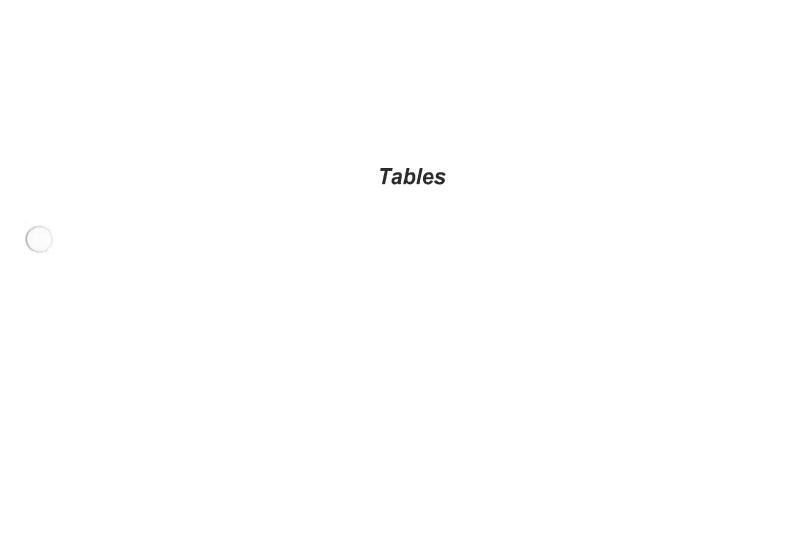


Table 3-1
Data Quality Objectives Design Summary

Project Objective	Data Needs	Analytes	DQO Statements
Identify areas and quantities of PCB contamination in Building 3 that will be included in a subsequent remedial action.	Horizontal and vertical extent of PCB contamination in concrete (floors, columns and walls), waste material, and soil at concentrations equivalent to or exceeding the action level.	PCBs	Collect data of sufficient quality to detect PCB concentrations at least 12.5 ppm. Collect data of sufficient quantity to identify areas within the building exceeding the 50 ppm action level.
Verify that oil staining is a reliable indicator for identifying PCB contamination in basement soils.	Concentrations of PCBs in soil.	PCBs	Collect data of sufficient quantity to detect PCB concentrations greater than the 50 ppm action level.
Pre-determine the waste characteristics of Building 3 concrete, waste material (Chip Chute waste pile and catch basins), and soil (basement floor and outside adjacent to Chip Chute) for potential removal and off-site disposal during a subsequent remedial action.	Representative concentrations of PCBs, RCRA hazardous waste constituents, and petroleum hydrocarbons.	PCBs Metals SVOCs GROs/DROs	Collect data of sufficient quality to detect PCB concentrations at least 12.5 ppm, metals and SVOCs at RCRA TCLP limits, and TPH/HEM at reporting limit (500 mg/kg). Collect data of sufficient quantity to be representative of building materials (concrete, waste, and soil) to potentially be disposed.
Pre-assess the health and safety concerns (i.e. personnel exposure) associated with Building 3 concrete, waste, and soils to support planning for a subsequent remedial action and building demolition.	Maximum concentration of contaminants (PCBs, etc.) expected to be present in building materials, and thus, present in dust generated during remediation and demolition activities.	PCBs Metals SVOCs	Collect data of sufficient quality to detect PCBs, metals, and SVOCs at concentrations at or greater than OSHA permissible exposure limits. Collect data of sufficient quantity to identify maximum contaminant levels to be encountered during remediation.
Assess personnel exposure to silica from potential dust-generating activities during the Building 3 sampling effort.	Concentration of airborne crystalline silica present in concrete dust generated during sampling and sample processing activities.	Silica	Collect data of sufficient quality to detect crystalline silica at OSHA PEL (10 mg/m³). Collect data of sufficient quantity to assess "worst case" dust environments.
Characterize IDW (decontamination water) from the Building 3 sampling effort to determine proper disposal methods.	Representative concentrations of PCBs and RCRA hazardous waste constituents.	PCBs Metals SVOCs	Collect data of sufficient quality to detect contaminant concentrations at least equivalent to water quality standards. (For PCBs – 0.5 µg/L). Collect data of sufficient quantity to be representative of decontamination water to be disposed or discharged to POTW (if discharge/water quality standards are met).

QAPP Tables - Rev 1

Table 4–1
Summary of Sampling and Analysis Program

Sampling Purpose	Media Type	Analytical Parameters	Extraction/Prep and Anal. Methods	Est. Number of Primary Samples (excluding QA/QC)
PCB Identification	Concrete	PCBs	SW-846 3540 or 3541/8082	464 [706]
	Soil	PCBs	SW-846 3540 or 3541/8082	92
	Waste	PCBs	SW-846 3540 or 3541/8082	11
Verification of Oil-Staining Selection Criteria	Soil	PCBs	SW-846 3540C/8082	4
Remediation Waste Pre- Determination	Concrete	TCLP SVOCs TCLP Metals	SW-846 1311/3580A/8270C SW-846 1311/6010B SW-846 1311/7471A	1
	Soil	TPH TCLP SVOCs TCLP Metals	SW-846 Method 8015B SW-846 1311/3580A/8270C SW-846 1311/6010B SW-846 1311/7471A	2
	Waste	TPH TCLP SVOCs TCLP Metals TPH	SW-846 8015B SW-846 1311/3580A/8270C SW-846 1311/6010B SW-846 1311/7471A SW-846 8015B	2
Health and Safety Pre-Assessment	Concrete	Total SVOCs Total Metals	SW-846 3540C/8270C SW-846 3050B/6010B SW-846 7471A/6010B	5
	Soil	Total SVOCs Total Metals	SW-846 3540C/8270C SW-846 3050B/6010B SW-846 7471A/6010B	3
	Waste	Total SVOCs Total Metals	SW-846 3540C/8270C SW-846 3050B/6010B SW-846 7471A/6010B	2
Health and Safety Monitoring	Air (Filter Cassette)	Total Respirable Dust Silica	NIOSH 0600 NIOSH 7500	5 est.

Sampling Purpose	Media Type	Analytical Parameters	Extraction/Prep and Anal. Methods	Est. Number of Primary Samples (excluding QA/QC)
IDW Characterization	Water	PCBs Total SVOCs Total Metals	SW-846 3510C/8082 SW-846 625/3510C/8270C SW-846 3010A/6010B	5 est.
			SW-846 7470A/6010B SW-846 3005A/6010B	

^[] Estimated maximum number of samples.

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Table 5-1
Container, Sample Volume, Preservation and Holding Time Requirements for
Concrete Samples

Analytical Group	Container(s)	Minimum Sample Size	Preservative	Holding Time
PCBs	One 4-oz glass jar with Teflon®-lined cap	10 g	N/A	14 days
Total SVOCs	One 4-oz glass jar with Teflon®-lined cap	50 g	Cool, 4±2°C	7 days
TCLP SVOCs	One 8-oz glass jar with Teflon®-lined cap	200 g	Cool, 4±2°C	14 days
Total Metals	One 4-oz glass jar	10 g	Cool, 4±2°C	180 days; 28 days for Hg
TCLP Metals	One 8-oz glass jar	200 g	Cool, 4±2°C	14 days
GRO/DRO	One 4-oz glass jar	50 g		14 days (OA1) 7 days (OA2)

Note: For every 20 field samples, and if the laboratory requires the extra volume to analyze MS/MSD samples, the minimum required sample volume will be tripled.

Table 5-2
Container, Sample Volume, Preservation, and Holding Time Requirements for Soil and
Waste Samples

Analytical Group	Container(s)	Minimum Sample Size	Preservative	Holding Time
PCBs	One 4-oz glass jar	125 g	N/A	14 days
Total SVOCs	One 4-oz glass jar with Teflon®-lined cap	50 g	Cool, 4±2°C	7 days
TCLP SVOCs	One 8-oz glass jar with Teflon®-lined cap	200 g	Cool, 4±2°C	14 days
Total Metals	One 4-oz glass jar	10 g	Cool, 4±2°C	180 days; 28 days for Hg
TCLP Metals	One 8-oz glass jar	200 g	Cool, 4±2°C	14 days
GRO/DRO	One 4-oz glass jar	50 g	Cool, 4±2°C	14 days (OA1) 7 days (OA2)

Note: For every 20 field samples, and if the laboratory requires the extra volume to analyze MS/MSD samples, the minimum required sample volume will be tripled.

Table 5-3
Container, Sample Volume, Preservation, and Holding Time Requirements for Water Samples

Analytical Group	Container(s)	Minimum Sample Size	Preservative	Holding Time
PCBs	Two 1-L amber glass jar with Teflon®-lined cap	2,000 ml	Cool, 4±2°C	7 days
Total SVOCs	Two 1-L or 1 ½-gal. amber glass bottle with Teflon®-lined cap	2,000 mL	Cool, 4±2°C	7 days
Total Metals	One 1-L plastic or glass container	300 mL	HNO ₃ to pH ≤2; Cool, 4±2°C	180 days; 28 days for Hg

Table 6-1
Collection Frequency for Field QA/QC Samples

QA/QC Sample Type	Frequency of Collection
Field Duplicates	10 % of total field samples
USACE Split Samples	10 % of total field samples
MS/MSD Samples	5 % of total field samples
Equipment Rinsate Blanks	5% of total field samples

Table 6-2
Estimated Number of Primary Samples and Field QA/QC Samples

Sampling Purpose	Media Type	Analytical Parameters	No. of Primary Samples	No. of Field Duplicates	No. of USACE Splits	No. of Equip. Rinsates*	No. of MS/MSD **
PCB	Concrete	PCBs	464	47	47	24	24
Identification		_	[706]	[71]	[71]	[36]	[36]
	Soil	PCBs	92	10	10	5	5
	Waste Pile	PCBs	11	2	2	1	1
Verification Oil- Staining Selection Criteria	Soil	PCBs	4	0	0	0	0
Remediation Waste Pre- Determination	Concrete	TCLP SVOCs TCLP Metals TPH	1	0	0	0	0
	Soil	TCLP SVOCs TCLP Metals TPH	2	1	1	0	0
	Waste	TCLP SVOCs TCLP Metals TPH	2	0	0	0	0
Health and Safety Pre-	Concrete	Total SVOCs Total Metals	5	1	1	0	0
Assessment	Soil	Total SVOCs Total Metals	3	0	0	0	0
	Waste	Total SVOCs Total Metals	2	0	0	0	0
IDW Characterization	Water	PCBs Total SVOCs Total metals	5	0	0	0	0
	TOTALS		591 [833]	61 [85]	61 [85]	30 [42]	30 [42]

^{*} Equipment rinsates are not necessary for remediation waste pre-determination samples and health and safety pre-assessment samples since they are collected at the same time as the corresponding PCB samples, using the same equipment.

^{**} If additional sample volume is needed by analytical laboratory.

^[] Estimated maximum number of samples.

Table 7-1
Reporting Limits for Method 8082 (PCBs)

Analyte	Concrete, Soil, Waste		W	ater
	RL*	Units	RL	Units
PCB – 1016	1	mg/kg	0.5	μg/L
PCB – 1221	1	mg/kg	0.5	μg/L
PCB – 1232	1	mg/kg	0.5	μg/L
PCB – 1242	1	mg/kg	0.5	μg/L
PCB – 1248	1	mg/kg	0.5	μg/L
PCB – 1254	1	mg/kg	0.5	μg/L
PCB - 1260	1	mg/kg	0.5	μg/L

^{*} Project-specific RL – provides a factor of safety to ensure that minimum sensitivity does not exceed 12.5 ppm.

Table 7-2
Precision and Accuracy Limits for Method 8082 (PCBs)

Analyte	Concrete, Soil, Waste		Wa	ter
	Accuracy (%Recovery)	Presision (RPD)	Accuracy (%Recovery)	Precision (RPD)
PCB - 1016	60 – 130	≤20	55 –115	≤20
PCB - 1260	55 - 145	≤20	60 -120	≤25

Table 7-3
Reporting Limits for Method 8270C (SVOCs)

Analyte		Water	So	il
	RL	Units	RL	Units
1,2,4-Trichlorobenzene	10	μg/L	330	μg/kg
1,2-Dichlorobenzene	10	μg/L	330	μg/kg
1,3-Dichlorobenzene	10	μg/L	330	μg/kg
1,4-Dichlorobenzene	10	μg/L	330	μg/kg
2,4-Dinitrotoluene	10	μg/L	330	μg/kg
2,6-Dinitrotoluene	10	μg/L	330	μg/kg
2-Chloronaphthalene	10	μg/L	330	μg/kg
2-Methylnaphthalene	10	μg/L	330	μg/kg
2-Nitroaniline	50	μg/L	1700	μg/kg
3-Nitroaniline	50	μg/L	1700	μg/kg
3,3'-Dichlorobenzidine	20	μg/L	700	μg/kg
3=4-Bromophenyl phenyl ether	10	μg/L	330	μg/kg
4-Chloroaniline	20	μg/L	700	π δ/ к δ
4-Chlorophenyl phenyl ether	10	μg/L	330	μg/kg
4-Nitroaniline	50	μg/L	1700	μg/kg
Acenaphthylene	10	μg/L	330	μg/kg
Acenaphthene	10	μg/L	330	μg/kg
Anthracene	10	μg/L	330	μg/kg
Benz (a) anthracene	10	μg/L	330	μg/kg
Benzo (a) pyrene	10	μg/L	330	μg/kg
Benzo (b) fluoranthene	10	μg/L	330	μg/kg
Benzo (g,h,i) perylene	10	μg/L	330	μg/kg
Benzyl alcohol	20	μg/L	700	μg/kg
Bis (2-chlorethoxy) methane	10	μg/L	330	μg/kg
Bis (2-chlorothyl) ether	10	μg/L	330	μg/kg
Bis (2-chloroisopropyl) ether	10	μg/L	330	μg/kg
Bis (2-ethylhexyl) phthalate	10	μg/L	330	μg/kg

Analyte		Water	S	oil
	RL	Units	RL	Units
Butyl benzylphthalate	10	μg/L	330	μg/kg
Chrysene	10	μg/L	330	μg/kg
Di-n-butylphthalate	10	μg/L	330	μg/kg
Di-n-octylphthalate	10	μg/L	. 330	µg/kg
Dibenz (a,h) anthracene	10	μg/L	330	μg/kg
Dibenzofuran	10	μg/L	330	μg/kg
Diethyl phthalate	10	μg/L	330	μg/kg
Dimethyl phthalate	10	μg/L	330	μg/kg
Fluoranthene	10	μg/L	330	μg/kg
Fluorene	10	μg/L	330	μg/kg
Hexachlorobenzene	10	μg/L	330	μg/kg
Hexachlorobutadiene	10	μg/L	330	μg/kg
Hexachlorocyclopentadiene	10	μg/L	330	μg/kg
Hexachloroethane	10	μg/L	330	μg/kg
Indeno (1,2,3-cd) pyrene	10	μg/L	330	μg/kg
Isophorone .	10	μg/L	330	μg/kg
n-Nitrosodimethylamine	10	μg/L	330	μg/kg
n-Nitrosodiphenylamine	10	μg/L	330	μg/kg
n-Nitrosodi-n-propylamine	10	μg/L	330	μg/kg
Naphthalene	10	μg/L	330	μg/kg
Nitrobenzene	10	μg/L	330	μg/kg
Phenanthrene	10	μg/L	330	μg/kg
Рутепс	10	μg/L	330	μg/kg

Table 7-4
Precision and Accuracy Limits for Method 8270C (SVOCs)

Analyte	Wa	iter	So	oil
Base/Neutral Fraction Compounds	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)	Precision (RDP)
1,2,4-Trichlorobenzene	40-145	≤20	30-150	≤30
1,2-Dichlorobenzene	40-155	≤20	30-130	≤30
1,3-Dichlorobenzene	35-125	≤20	35-135	≤30
1,4-Dichlorobenzene	30-125	≤20	25-135	≤30
2,4-Dinitrotoluene	35-140	≤20	25-149	≤30
2,6-Dinitrotoluene	50-125	≤20	40-135	≤30
2-Chloronaphthalene	55-125	≤20	45-135	≤30
2-Methylnaphthalene	40-125	≤20	30-135	≤30
2-Nitroaniline	45-125	≤20	45-135	≤30
3-Nitroaniline	45-125	≤20	25-175	≤30
3,3'-Dichlorobenzidine	25-175	≤20	25-175	≤30
4-Bromophenyl phenyl ether	50-125	≤20	40-135	≤30
4-Chloroaniline	40-135	≤20	35-135	≤30
4-Chlorophenyl phenyl ether	50-130	≤20	40-140	≤30
4-Nitroaniline	35-140	≤20	25-150	≤30
Acenaphthylene	45-125	≤20	35-135	≤30
Acenaphthene	45-155	≤20	35-135	≤30
Anthracene	40-165	≤20	35-175	≤30
Benz (a) anthracene	50-135	≤20	40-140	≤30
Benzo (a) pyrene	40-125	≤20	30-130	≤30
Benzo (b) fluoranthene	35-125	≤20	25-135	≤30
Benzo (g,h,i) perylene	30-150	≤20	25-155	≤30
Benzyl alcohol	30-125	≤20	25-135	≤30
Bis (2-chlorethoxy) methane	45-125	≤20	35-135	≤30
Bis (2-chlorothyl) ether	40-125	≤20	30-135	≤30

Analyte	Wa	ter	Se	oil
Base/Neutral Fraction Compounds	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)	Precision (RDP)
Bis (2-chloroisopropyl) ether	30-135	≤20	25-175	≤30
Bis (2-ethylhexyl) phthalate	30-130	≤20	25-135	≤30
Butyl benzylphthalate	20-125	≤20	25-135	≤30
Chrysene	55-135	≤20	45-140	≤30
Di-n-butylphthalate	30-125	≤20	25-135	≤30
Di-n-octylphthalate	35-130	≤20	25-135	≤30
Dibenz (a,h) anthracene	50-125	≤20	40-135	≤30
Dibenzofuran	50-125	≤20	40-135	≤30
Diethyl phthalate	35-125	≤20	25-135	≤30
Dimethyl phthalate	25-125	≤20	25-175	≤30
Fluoranthene	45-125	≤20	35-135	≤30
Fluorene	45-135	≤20	35-145	≤30
Hexachlorobenzene	45-135	≤20	35-140	≤30
Hexachlorobutadiene	25-125	≤20	25-135	≤30
Hexachlorocyclopentadiene	20-125	≤20	30-135	≤30
Hexachloroethane	25-150	≤20	25-165	≤30
Indeno (1,2,3-cd) pyrene	25-160	≤20	25-175	≤30
Isophorone	25-175	≤20	25-175	≤30
n-Nitrosodimethylamine	25-125	≤20	25-175	≤30
n-Nitrosodiphenylamine	25-125	≤20	25-135	≤30
n-Nitrosodi-n-propylamine	35-125	≤20	25-135	≤30
Naphthalene	45-125	≤20	40-135	≤30
Nitrobenzene	40-130	≤20	35-140	≤30
Phenanthrene	50-120	≤20	40-135	≤30
Pyrene	45-145	≤20	35-145	≤30

Table 7-5
Reporting Limits for Method 6010B (Metals)

Analyte	Wa	ater	5	Soil
	RL	Units	RL	Units
Aluminum	200	μg/L	20	mg/kg
Antimony	50	μg/L	5	mg/kg
Arsenic	10	μg/L	1	mg/kg
Barium	50	μg/L	5	mg/kg
Beryllium	5	μg/L	0.5	mg/kg
Cadmium	5	μg/L	0.5	mg/kg
Calcium	1000	μg/L	100	mg/kg
Chromium	10	μg/L	1	mg/kg
Cobalt	10	μg/L	11	mg/kg
Copper	10	μg/L	1	mg/kg
Iron	100	μg/L	10	mg/kg
Lead	3	μg/L	0.3	mg/kg
Magnesium	100	μg/L	10	mg/kg
Manganese	5	μg/L	0.5	mg/kg
Nickel	10	μg/L	1	mg/kg
Potassium	500	μg/L	50	mg/kg
Selenium	10	μg/L	1	mg/kg
Silver	10	μg/L	1	mg/kg
Sodium	1000	μg/L	100	mg/kg
Thallium	10	μg/L	1	mg/kg
Vanadium	10	μg/L	1	mg/kg
Zinc	10	μg/L	1	mg/kg

Table 7-6
Precision and Accuracy Limits for Method 6010B (Metals)

Analyte	Wa	Water		oil
	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)
Aluminum	75-125	±20	75-125	±35
Antimony	75-125	±20	75-125	±35
Arsenic	75-125	±20	75-125	±35
Barium	75-125	±20	75-125	±35
Beryllium	75-125	±20	75-125	±35
Cadmium	75-125	±20	75-125	±35
Calcium	75-125	±20	75-125	±35
Chromium	75-125	±20	75-125	±35
Copper	75-125	±20	75-125	±35
Iron	75-125	±20	75-125	±35
Lead	75-125	±20	75-125	±35
Magnesium	75-125	±20	75-125	±35
Manganese	75-125	±20	75-125	±35
Nickel	75-125	±20	75-125	±35
Potassium	75-125	±20	75-125	±35
Selenium	75-125	±20	75-125	±35
Silver	75-125	±20	75-125	±35
Sodium	75-125	±20	75-125	±35
Thallium	75-125	±20	75-125	±35
Vanadium	75-125	±20	75-125	±35
Zinc	75-125	±20	75-125	±35

Table 7-7
Reporting Limits and Precision and Accuracy Limits for Method 7470A/7471A (Mercury)

Reporting Limits

Analyte	Water (7470A)		Soil (7	/471A)
	RL	Units	RL	Units
Mercury	1.0	μg/L	0.1	mg/kg

Precision and Accuracy Limits

Analyte	Water (7470A)		Soil (7	/471A)
	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)
Mercury	70-130	≤20	70-130	≤35

Table 7-8
Reporting Limits and Precision and Accuracy Limits for Method 8015B(M) (GROs/DROs)

Reporting Limits

Media	GRO - Total Purgeable Hydrocarbons (OA1)		DRO - Semivolatile Petroleum Hydrocarbons (OA2)	
	RL Units		RL	Units
Soil	100	μg/kg	5	mg/kg

Precision and Accuracy Limits

Media	GRO - Total Purgeable Hydrocarbons (OA1) Accuracy* Precision (%Recovery) (RPD)		DRO - Semivolatile Petroleum Hydrocarbons (OA2)	
			Accuracy* (%Recovery)	Precision (RPD)
Soil	70-130	≤35	60 -140	≤35

^{*} Based on LCS/LCSD

Table 8-1
Calibration and QC Check Requirements for Method 8082 (PCBs)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Five-point initial calibration for AR-1016 and AR-1260, plus single standard of each	Prior to sample analysis	Linear - mean RSD for all analytes #20% with no individual analyte >30% RSD	Correct problem, then repeat initial calibration
of the remaining five Aroclors.		Linear-least squares regression r∃0.995	
For Congener analysis, calibrate for all congeners with fivelevel standard curve		Acceptable Response Factor for single standard compound	
Peak Quantitation	For each Aroclor	Use of a minimum of three peaks (preferably five) peaks per compound for quantitation	The mean response or Calibration Factor, the Standard Deviation, and %Relative Standard Deviation must meet Method 8000 requirements
Retention time window calculated for each analyte		Chosen appropriately to alleviate false positive and false negative results*	Correct problem, then reanalyze all samples analyzed since the last retention time check
Initial Calibration Verification	Every 12 hours following initial calibration and before any sample analysis	All analytes within ∀15% of expected value.	Correct problem, then repeat initial calibration
Continuing Calibration Verification	After every 10 samples and at the end of the analysis sequence	All analytes within ∀15% of expected value (85% - 115% R)	Correct problem then repeat initial calibration
Method Blank (MB)	One MB per	No analytes detected	Qualify all affected

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
	analytical batch; not to exceed 1 MB per twenty field samples	∃Reporting Limit	compounds for samples in analytical batch
Laboratory Control Sample (LCS) for all analytes	One LCS per analytical batch	All spiked analytes recovery within 80-120% of certified value	Reprep LCS and all samples in the affected analytical batch
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	One MS/MSD set per 20 client samples	Sample spiked minimally with AR- 1016 and- 1260 and recovered within lab established control limits	Qualify all samples in analytical batch estimated for out-of control analytes
Duplicate Sample Analysis	One duplicate sample analysis per set of 20 client field samples.	All positive detects analytes should agree within ∀25% D	Qualify the original and duplicate hits as estimated; out-of- control
Surrogate Spikes	Minimum one added to every sample, blank, QC, and std.	Recovered within lab established control limits	Rerun to confirm matrix interference; re-extract if necessary
Internal Standards	Added to every sample and all QC solutions if determination of congeners is performed	Refer to Method 8000 for criteria	

^{*} Laboratory SOPs will be reviewed to ensure that acceptable manual integration is practiced.

Table 8-2
Calibration and QC Check Requirements for Method 8270C (SVOCs)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Five-point initial calibration for all analytes	Prior to sample analysis	SPCCs average RF ≥0.50 and %RSD for RFs for CCCs ≤30% and one of the below:	Correct problem, then repeat initial calibration
		1) Linear-mean RSD ≤15% for all analytes; no individual analyte _30% RSD	
		2) Linear-least squares regresssion r≥0.995	
		3) Non-linear - COD ≥0.990	
Second-source calibration verification	Once per five-point calibration	All analytes within ±25% of expected results	Correct problem then repeat initial calibration
Retention time window calculated for each analyte	Each Sample	Relative Retention Time (RRT) of the analyte within ±0.06 units of the RRT	Correct problem, then reanalyze all samples analyzed since the last retention time check
Surrogate Spikes	Minimum one added to every sample, blank, QC and std.	All surrogate spike recovery within 60 – 130% for base/neutral compounds and 35 – 135% for acids	Re-extract and reanalyze affected sample
Continuing Calibration Verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥0.050; and CCCs ≤20% difference or drift	Correct problem then repeat initial calibration

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	
Internal Standards	6 per each sample	Retention time ±30 seconds from the retention time of the mid-point std. of the ICAL	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of all	
		EICP area within -50% to +100% of the ICAL mid point std.	samples out-of control	
Method Blank (MB)	One MB per analytical batch	No analytes detected ≥Reporting Limit (RL)	Qualify affected compounds for associated samples with B to indicate blank contamination	
Laboratory Control Sample (LCS) for all analytes	One LCS per analytical batch	Recover all spiked compounds within laboratory established control limits	Re-extract entire sample batch and associated QC and rerun	
Check of mass spectal ion intensities using DFTPP Tune	Prior to initial calibration and calibration verification	All mass ion abundances as per method 8270C; expanded criteria allowed within CLP protocol	Inspect mass spectrometer and GC for malfunctions; rerun DFTPP and retune hardware	
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	One MS/MSD set per 20 client samples	Recover all spiked compounds within laboratory established control limits	Qualify affected compounds for associated samples to indicate estimated concentrations	

Table 8-3
Calibration and QC Check Requirements for Method 6010B (Metals)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial calibration for all analytes consisting of a minimum blank and high standard	Daily prior to sample analysis	None	None
Initial Calibration Verification (second- source)	Daily after initial calibration	All analytes within ±10% of expected results	Correct problem then repeat initial calibration
Calibration Blank	After every calibration verification	No analytes detected ≥Reporting Limit (RL)	Correct problem, then reanalyze calibration blank and previous 10 samples
Calibration Verification (Instrument Check Standard)	After every 10 samples and at the end of the analysis sequence	All analytes with ±10% of expected results and RSD of replicate integrations <5%	Repeat calibration and reanalyze all samples since successful calibration
Interference Check Standard (ICS)	At the beginning of the an analytical run	All analytes with ±20% of expected results	Terminate analysis; correct problem; reanalyze ICS; reanalyze all affected samples
Method Blank (MB)	One MB per analytical batch	No analytes detected ≥Reporting Limit (RL)	None; qualify associated samples according to 5X rule
Laboratory Control Sample (LCS) for all analytes	One LCS per analytical batch	Recover all analytes within 80- 120% of true value	Re-digest entire sample for re-analysis of out of control analyte
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	One MS/MSD set per 20 client samples	Recover all analytes within 75- 125% of spoiked	Qualify all associated sample as estimated for out of control

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Duplicate (MSD)	samples	value	analyte
Serial Dilution Test	One sample per sample digestion batch	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition
Post Digestion Spike Addition	When serial dilution test fails.	Recovery within 75-125% of expected results	Correct problem then reanalyze post digestion spike addition

Table 8-4
Calibration and QC Check Requirements for Method 7470A/7471A (Mercury)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial multipoint calibration (minimum 5 standards and a blank	Daily prior to sample analysis	Correlation Coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration
Second Source Calibration Verification	Once per initial calibration	Analytes within ±10% of expected results	Correct problem then repeat initial calibration
Calibration Blank	After every calibration verifications	Analytes detected ≥Reporting Limit (RL)	Correct problem, then reanalyze calibration blank and previous 10 samples
Calibration Verification (Instrument Check Standard)	After every 10 samples and at the end of the analysis sequence	Analytes with ±20% of expected results	Repeat calibration and reanalyze all samples since successful calibration
Method Blank (MB)	One MB per analytical batch	No analytes detected ≥Reporting Limit (RL)	Qualify associated using the 5X rule for blank contamination
Laboratory Control Sample (LCS) for all analytes	One LCS per analytical batch	Recover within 80- 120% of true	Re-digest entire sample batch
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	One MS/MSD set per 20 client samples	Recover within 70-130% and agree within 20% for waters and 35% for soils	Qualify associated samples' results as estimated

Table 10-1
Summary of Typical Laboratory Preventative Maintenance Procedures

Instrument	Activity	Frequency
Gas Chromatograph / Mass Spectrometer Semivolatile Organic Compounds (GC/MS)	Clean mass spectrometer source Change septum Change liners Check carrier gas Change carrier gas Change in-line filters Remove first foot of column Bake out column Check system for gas leaks Sylonize injection port liners	When tuning criteria cannot be achieved Daily Daily Daily When pressure ≤500psi Quarterly, as needed To improve chromatography To improve chromatography At each column change Every liner
High Performance Liquid Chromatograph (HPLC)	Check / change degas gases Check / change guard column Check / replace pre-column frits Monitor UV lamp intensity Replace Column Check flows	Daily Weekly Weekly As needed As needed Weekly
Cold Vapor Atomic Absorption Spectrophotomer (CVAAS)	Clean optical windows Check plumbing connections Check gases Change drying tube Change tubing	Daily Daily Daily Daily Weekly
Inductively Coupled Plasma Optical Emission Spectrometer (ICP or ICPOES)	Check gas flow Clean nebulizer Check torch Change tubing Check optics	Daily Weekly Weekly, or as needed Weekly, or as needed Annual service contract
UV/Visible Spectrophotometer (UV/Vis)	Clean spectrophotometer windows Change spectrophotometer cuvettes Check autosampler and tubing Check filters	Daily Daily Daily Monthly
Ovens	Temperature monitoring	Once daily
Refrigerators	Temperature monitoring	Once daily
Analytical Balances	Check pans and compartment Check alignment and calibration Cleaning/ Service	Prior to use Before every use Semi-anually

Table 12-1
Calculations for Data Quality Indicators

Statistic	Symbol	Formula	Definition	Use
Mean	$\frac{1}{x}$	$\left(\begin{array}{c} \sum_{i=1}^{n} x_{i} \\ \hline n \end{array}\right)$	Measure of central tendency	Used to determine the average value of multiple measurements
Standard Deviation	S	$\sqrt{\left(\frac{\sum (x_i - \overline{x})^2}{n-1}\right)}$	Measure of the relative scatter of the data	Used in calculating variation of measurements
Relative Standard Deviation	RSD	$(\sqrt[3]{x}) \times 100$	Relative standard deviation adjusts for the magnitude of observations	Used to assess the precision parameter for replicate results
Percent Difference	%D	$\left(\frac{x_1 - x_2}{x_1}\right) \times 100$	Measure of the difference between two observations	Used to assess the accuracy parameter
Relative Percent Difference	RPD	$\left(\frac{x_1 - x_2}{\left(x_1 + x_2\right) \div 2}\right) \times 100$	Measure of variability that adjusts for the magnitude of observations	Used to assess the analytical precision of duplicate measurements
Percent Recovery	% R	$\left(\frac{x_{measured}}{x_{true}}\right) \times 100$	Recovery of spiked compounds in control sample (LCS)	Used to assess the accuracy parameter
Percent Recovery	% R	$\frac{\left(x_s - x_u\right)}{x_a} \times 100$ where:	Recovery of spiked compounds in the sample matrix	Used to assess matrix effects and precision between the MS and MSD
		x_s is the value of the spiked sample, x_u is the value of the unspiked sample, x_a is amount spiked into the sample		

Table 13-1
Summary of Analytical Data Deliverable Requirements

Method requirements	Deliverables
Requirements for all methods:	
- Holding time information and methods requested	Signed chain-of-custody forms
- Discussion of laboratory analysis, including any laboratory problems	Case narratives
Organics: GC/MS analysis	
- Sample results, including TICs	CLP Form 1 or equivalent
- Surrogate recoveries	CLP Form 2 or equivalent
- Matrix spike/spike duplicate data	CLP Form 3 or equivalent
- Method blank data	CLP Form 4 or equivalent
- GC/MS tune	CLP Form 5 or equivalent
- GC/MS initial calibration data	CLP Form 6 or equivalent
- GC/MS continuing calibration data	CLP Form 7 or equivalent
- GC/MS internal standard area data	CLP Form 8 or equivalent
Organics: GC analysis	
- Sample results	CLP Form 1 or equivalent
- Surrogate recoveries	CLP Form 2 or equivalent
- Matrix spike/spike duplicate data	CLP Form 3 or equivalent
- Method blank data	CLP Form 4 or equivalent
- Initial calibration data	CLP Form 6 or equivalent
- If calibration factors are used	A form listing each analyte, the concentration
	of each standard, the relative calibration factor,
	the mean calibration factor, and %RSD
- Calibration curve if used	Calibration curve and correlation coefficient
 Continuing calibration data 	CLP Form 9 or equivalent
- Positive identification (second column	CLP Form 10 or equivalent
confirmation)	
Metals	
- Sample results	CLP Form 1 or equivalent
- Initial and continuing calibration	CLP Form 2 or equivalent, dates of analyses
	and calibration curve, and the correlation
- Method blank	coefficient factor
- ICP interference check sample	CLP Form 3 or equivalent and dates of analyses
- Spike sample recovery	CLP Form 4 or equivalent and dates of analyses
- Postdigestion spike sample recovery for ICP	CLP Form 5A or equivalent
metals	CLP Form 5B or equivalent

Method requirements	Deliverables
- Postdigestion spike for GFAA	CLP Form 5B or equivalent
- Duplicates	CLP Form 6 or equivalent
- LCS	CLP Form 7 or equivalent that includes
	acceptable range or window
- Standard additions (when implemented)	CLP Form 8 or equivalent
- Holding times	CLP Form 13 or equivalent
- Run log	CLP Form 14 or equivalent
_	
Wet Chemistry	
- Sample results	Report result
- Matrix spike recovery	%Recovery
- Matrix spike duplicate or duplicate	%Recovery and %RPD
- Method blank	Report results
- Initial calibration	Calibration curve and correlation coefficient
- Continuing calibration check	Recovery and % difference
- LCS	LCS result and control criteria
- Run log	Copy of run log

Appendix A NIOSH Methods for Respirable Dust and Silica Analysis

PARTICULATES NOT OTHERWISE REGULATED, RESPIRABLE

DEFINITION: aerosol collected by sampler with 4-um median cut point

CAS: None

RTECS: None

0600

EVALUATION: FULL

Issue 1: 15 February 1984

Issue 3: 15 January 1998

OSHA: 5 mg/m³ NIOSH: no REL ACGIH: 3 mg/m3

METHOD: 0600, Issue 3

PROPERTIES:

contains no asbestos and quartz less than 1%; penetrates non-ciliated portions of respiratory

system

SYNONYMS:

nuisance dusts; particulates not otherwise classified

SAMPLING

GRAVIMETRIC (FILTER WEIGHT)

SAMPLER: CYCLONE + FILTER

(10-mm nylon cyclone, Higgins-Dewell [HD] cyclone, or Aluminum cyclone + tared 5-µm

PVC membrane)

20 L @ 5 mg/m³

ANALYTE:

mass of respirable dust fraction

nylon cyclone: 1.7 L/min HD cyclone: 2.2 L/min

Al cyclone: 2.5 L/min

VOL-MIN: -MAX:

FLOW RATE:

400 L

SHIPMENT: routine

SAMPLE

STABILITY:

stable

BLANKS:

2 to 10 field blanks per set

TECHNIQUE:

MEASUREMENT

BALANCE:

0.001 mg sensitivity; use same balance before and after sample collection

CALIBRATION:

National Institute of Standards and Technology Class S-1.1 or ASTM Class 1

weights

RANGE:

0.1 to 2 mg per sample

ESTIMATED LOD: 0.03 mg per sample

PRECISION:

<10 µg with 0.001 mg sensitivity balance; <70

µg with 0.01 mg sensitivity balance [3]

ACCURACY

RANGE STUDIED: 0.5 to 10 mg/m3 (lab and field)

BIAS:

dependent on dust size distribution [1]

OVERALL

PRECISION (\$,T):

dependent on size distribution [1,2]

ACCURACY:

dependent on size distribution [1]

APPLICABILITY: The working range is 0.5 to 10 mg/m³ for a 200-L air sample. The method measures the mass concentration of any non-volatile respirable dust. In addition to inert dusts [4], the method has been recommended for respirable coal dust. The method is biased in light of the recently adopted international definition of respirable dust, e.g., ≈ +7% bias for non-diesel, coal mine dust [5].

INTERFERENCES: Larger than respirable particles (over 10 µm) have been found in some cases by microscopic analysis of cyclone filters. Over-sized particles in samples are known to be caused by inverting the cyclone assembly. Heavy dust loadings, fibers, and watersaturated dusts also interfere with the cyclone's size-selective properties. The use of conductive samplers is recommended to minimize particle charge effects.

OTHER METHODS: This method is based on and replaces Sampling Data Sheet #29.02 [6].

EQUIPMENT:

- 1. Sampler:
 - a. Filter: 5.0-µm pore size, polyvinyl chloride filter or equivalent hydrophobic membrane filter supported by a cassette filter holder (preferably conductive).
 - b. Cyclone: 10-mm nylon(Mine Safety Appliance Co., Instrument Division, P. O. Box 427, Pittsburgh, PA 15230), Higgins-Dewell(BGI Inc., 58 Guinan St., Waltham, MA 02154)[7], aluminum cyclone (SKC Inc., 863 Valley View Road, Eighty Four, PA 15330), or equivalent.
- 2. Personal sampling pump, 1.7 L/min ± 5% for nylon cyclone, 2.2 L/min ± 5% for HD cyclone, or 2.5 L/min ± 5% for the Al cyclone with flexible connecting tubing.

NOTE: Pulsation in the pump flow must be within ± 20% of the mean flow.

- 3. Balance, analytical, with sensitivity of 0.001 mg.
- 4. Weights, NIST Class S-1.1, or ASTM Class 1.
- 5. Static neutralizer, e.g., Po-210; replace nine months after the production date.
- 6. Forceps (preferably nylon).
- 7. Environmental chamber or room for balance, e.g., 20°C ± 1°C and 50% ± 5% RH.

SPECIAL PRECAUTIONS: None.

PREPARATION OF SAMPLERS BEFORE SAMPLING:

- 1. Equilibrate the filters in an environmentally controlled weighing area or chamber for at least 2 h.
- 2. Weigh the filters in an environmentally controlled area or chamber. Record the filter tare weight, W (mg).
 - a. Zero the balance before each weighing.
 - b. Handle the filter with forceps (nylon forceps if further analyses will be done).
 - c. Pass the filter over an anti-static radiation source. Repeat this step if filter does not release easily from the forceps or if filter attracts balance pan. Static electricity can cause erroneous weight readings.
- 3. Assemblethe filters in the filter cassettes and close firmly so that leakage around the filter will not occur. Place a plug in each opening of the filter cassette.
- 4. Remove the cyclone's grit cap before use and inspect the cyclone interior. If the inside is visibly scored, discard this cyclone since the dust separation characteristics of the cyclone may be altered. Clean the interior of the cyclone to prevent reentrainment of large particles.
- 5. Assemble the sampler head. Check alignment of filter holder and cyclone in the sampling head to prevent leakage.

SAMPLING:

- 6. Calibrate each personal sampling pump to the appropriate flow rate with a representative sampler in line.
 - NOTE 1: Because of their inlet designs, nylon and aluminum cyclones are calibrated within a large vessel with inlet and outlet ports. The inlet is connected to a calibrator (e.g., a bubble meter). The cyclone outlet is connected to the outlet port within the vessel, and the vessel outlet is attached to the pump. See APPENDIX for alternate calibration procedure. (The calibrator can be connected directly to the HD cyclone.)
 - NOTE 2: Even if the flowrate shifts by a known amount between calibration and use, the nominal flowrates are used for concentration calculation because of a self-correction feature of the cyclones.
- 7. Sample 45 min to 8 h. Do not exceed 2 mg dust loading on the filter. Take 2 to 4 replicate samples for each batch of field samples for quality assurance on the sampling procedure (see Step 10).
 - NOTE: Do not allow the sampler assembly to be inverted at any time. Turning the cyclone to anything more than a horizontal orientation may deposit oversized material from the cyclone body onto the filter.

SAMPLE PREPARATION:

8. Remove the top and bottom plugs from the filter cassette. Equilibrate for at least 2 h in an environmentally controlled area or chamber.

CALIBRATION AND QUALITY CONTROL:

- 9. Zero the microbalance before all weighings. Use the same microbalance for weighing filters before and after sample collection. Calibrate the balance with National Institute of Standards and Technology Class S-1.1 or ASTM Class 1 weights.
- 10. The set of replicate field samples should be exposed to the same dust environment, either in a laboratory dust chamber [8] or in the field [9]. The quality control samples must be taken with the same equipment, procedures, and personnel used in the routine field samples. Calculate precision from these replicates and record relative standard deviation (9 on control charts. Take corrective action when the precision is out of control [8].

MEASUREMENT:

11. Weigh each filter, including field blanks. Record this post-sampling weight, W(mg), beside its corresponding tare weight. Record anything remarkable about a filter (e.g., visible particles, overloading, leakage, wet, torn, etc.).

CALCULATIONS:

12. Calculate the concentration of respirable particulate, C (mg/n), in the air volume sampled, V (L):

$$C = \frac{(W_2 - W_1) - (B_2 - B_1)}{V}$$
. 10³, mg/m^3

where: W_1 = tare weight of filter before sampling (mg)

 W_2 = post-sampling weight of sample-containing filter (mg)

 B_1 = mean tare weight of blank filters (mg).

 B_2 = mean post-sampling weight of blank filters (mg)

V = volume as sampled at the nominal flowrate (i.e., 1.7 L/min or 2.2 L/min)

EVALUATION OF METHOD:

1. Bias: In respirable dust measurements, the bias in a sample is calculated relative to the appropriate respirable dust convention. The theory for calculating bias was developed by Bartley and Breuer [10]. For this method, the bias, therefore, depends on the international convention for respirable dust, the cyclones' penetration curves, and the size distribution of the ambient dust. Based on measured penetration curves for non-pulsating flow [1], the bias in this method is shown in Figure 1.

For dust size distributions in the shaded region, the bias in this method lies within the \pm 0.10 criterion established by NIOSH for method validation. Bias larger than \pm 0.10 would, therefore, be expected for some workplace aerosols. However, bias within \pm 0.20 would be expected for dusts with geometric standard deviations greater than 2.0, which is the case in most workplaces.

Bias can also be caused in a cyclone by the pulsation of the personal sampling pump. Bartley, et al. [12] showed that cyclone samples with pulsating flow can have negative bias as large as 0.22 relative to samples with steady flow. The magnitude of the bias depends on the amplitude of the pulsation at the cyclone aperture and the dust size distribution. For pumps with instantaneous flow rates within 20% of the mean, the pulsation bias magnitude is less than 0.02 for most dust size distributions encountered in the workplace.

Electric charges on the dust and the cyclone will also cause bias. Briant and Moss [13] have found

electrostatic biases as large as -50%, and show that cyclones made with graphite-filled nylon eliminate the problem. Use of conductive samplers and filter cassettes(Omega Specialty Instrument Co., 4 Kidder Road, Chelmsford, MA 01824) is recommended.

2. Precision: The figure 0.068 mg quoted above for the precision is based on a study [3] of weighing procedures employed in the past by the Mine Safety and Health Administration (MSHA) in which filters are pre-weighed by the filter manufacturer and post-weighed by MSHA using balances readable to 0.010 mg. MSHA [14] has recently completed a study using a 0.001 mg balance for the post-weighing, indicating imprecision equal to 0.006 mg.

Imprecision equal to 0.010 mg was used for estimating the LOP dis based on specific suggestions [8] regarding filter weighing using a single 0.001 mg balance. This value is consistent with another study [15] of repeat filter weighings, although the actual attainable precision may depend strongly on the specific environment to which the filters are exposed between the two weighings.

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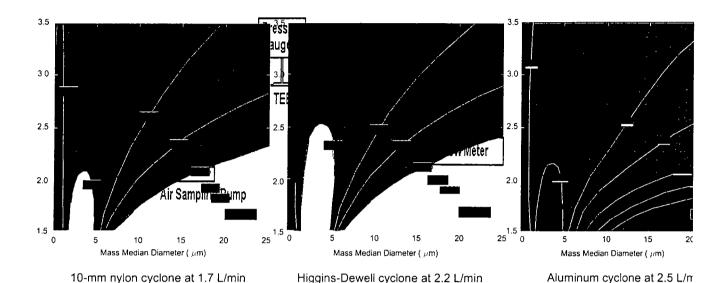


Figure 1. Bias of three cyclone types relative to the international respirable dust samp convention.

APPENDIX: Jarless Method for Calibration of Cyclone Assemblies

This procedure may be used in the field to calibrate an air sampling pump and a cyclone assembly without using the one-liter "calibration jar".

- (1) Connect the pump to a pressure gauge or water manometer and a light load (adjustable valve or 5-µm filter) equal to 2" to 5" H₂O with a "TEE" connector and flexible tubing. Connect other end of valve to an electronic bubble meter or standard bubble tube with flexible tubing (See Fig. 2.1).
 - NOTE: A light load can be a 5-µm filter and/or an adjustable alve. A heavy load can be several 0.8-µm filters and/or adjustable valve.
- (2) Adjust the pump to 1.7 L/min, asindicated on the bubble meter/tube, under the light load conditions (2" to 5" H₂O) as indicated on the pressure gauge or manometer.
- (3) Increase the load until the pressure gauge or water manometer indicates between 25" ara5" H₂O. Check the flow rate of the pump again. The flow rate should remain at 1.7 L/min ± 5%.
- (4) Replace the pressure gauge or water manometer and the electronic bubble meter or standard bubble tube with the cyclone having a clean filter installed (Fig. 2.2). If the loading caused by the cyclone assembly is between 2" and 5" HO, the calibration is complete and the pump and cyclone are ready for sampling.

Figure 2.1 Block Diagram of Pump/Load/Flow Meter Set-up.

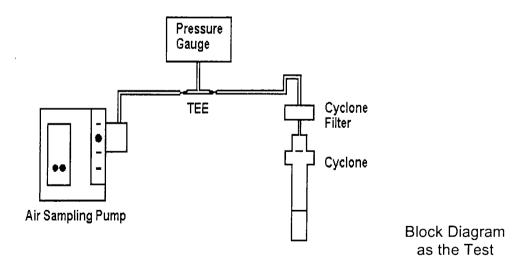


Figure 2.2. with Cyclone Load.

SILICA, CRYSTALLINE, by XRD

7500

SiO₂

MW: 60.08

CAS: 14808-60-7 (quartz)

RTECS: VV7330000

(quartz)

14464-46-1 (cristobalite)

VV7325000

(cristobalite)

15468-32-3 (tridymite)

VV7335000 (tridymite)

METHOD: 7500, Issue 3

EVALUATION: FULL

Issue 1: 15 August 1990

OSHA: quartz (respirable) 10 mg/m³/(%SiO₂+2);

Issue 3: 15 January 1998

solid; d 2.65 g/cm3 @ 0 °C; crystalline

NIOSH: 0.05 mg/m³; carcinogen ACGIH: quartz (respirable) 0.1 mg/m3

cristobalite and tridymite (respirable) 1/2 the above

transformations: quartz to tridymite @ 867 °C; tridymite to cristobalite @ 1470

cristobalite (respirable) 0.05 mg/m3

°C; α-quartz to ß-quartz @ 573 °C

tridymite (respirable) 0.05 mg/m3

SYNONYMS: free crystalline silica; silicon dioxide

SAMPLING

MEASUREMENT

crystalline SiO₂

monochromator

PRECISION (5.): 0.08 @ 0.05 to 0.2 mg per sample [1]

subtraction

X-RAY POWDER DIFFRACTION

muffle furnace or RF plasma asher

or dissolve in tetrahydrofuran

on 0.45-µm Ag membrane filter

Cu target X-ray tube, graphite

Optimize for intensity; 1° slit Slow step scan, 0.02°/10 sec

SAMPLER:

CYCLONE + FILTER

TECHNIQUE: (10-mm nylon cyclone, Higgins-Dewell

(HD) cyclone, or aluminum cyclone +

5-µm PVC membrane

FLOW RATE:

nylon cyclone: 1.7 L/min;

HD cyclone: 2.2 L/min; aluminum cyclone: 2.5 L/min

VOL-MIN: -MAX:

400 L 1000 L

SHIPMENT:

routine

SAMPLE

STABILITY:

stable

BLANKS:

2 to 10 per set (See step 12.g.)

BULK SAMPLE:

high-volume or settled dust; to identify interferences

CALIBRATION:

RANGE:

suspensions of silica in 2-propanol

Integrated intensity with background

ANALYTE:

REDEPOSIT:

ASH:

XRD:

0.02 to 2 mg SiO₂ per sample [2]

PROPERTIES:

ESTIMATED LOD: 0.005 mg SiO₂ per sample [2]

ACCURACY

RANGE STUDIED:

25 to 2500 µg/m³ [1] (800-L sample)

BIAS:

none known

OVERALL PRECISION $\hat{\mathbf{S}}_{rT}$): 0.09 (50 to 200 µg) [1]

ACCURACY:

± 18%

APPLICABILITY: The working range is 0.025 to 2.5 mg/m³ for an 800-L air sample.

INTERFERENCES: Micas, potash, feldspars, zircon, graphite, and aluminosilicates. See APPENDIX.

OTHER METHODS: This is similar to the method in the Criteria Document [3] and P&CAM 259 [4] which has been collaboratively tested [2]. This method is similar, except for sample collection, to S315 [5,6]. Method P&CAM 109 [7,8,9], which incorporates an internal standard, has been dropped. Colorimetry [10] is an alternate measurement procedure (used in Method 7601), as is IR spectrometry (Methods 7602 and 7603).

REAGENTS:

- 1. Silica Standards.
 - a. Quartz* (SRMs 1878 and 2679) and Cristobalite* (SRM 1879), available from Standard Reference Materials Program, Rm. 204, Bldg. 202, National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899.
 - b. Tridymite*, available from NIOSH, DPSE, ARDB, 4676 Columbia Pkwy., Cincinnati, OH 45226.
- 2. 2-Propanol, reagent grade.
- 3. Desiccant.
- 4. Glue or tape for securing Ag filters to XRD holders.
- Optional: tetrahydrofuran (THF)* (if LTA or muffle furnace are unavailable).
- Optional (if calcite present): 25% v/v concentrated hydrochloric acid (ACS reagent grade) in distilled water and 25-mm filters of PVC or cellulose ester with pore size of 1 µm or less.
 - * See SPECIAL PRECAUTIONS.

EQUIPMENT:

- 1. Sampler:
 - a. Filter: Polyvinyl chloride (PVC) filter, 37mm, 5.0-µm pore size supported with backuppad in a two-piece, 37-mm cassette filter holder (preferably, conductive) held together by tape or cellulose shrink band.
 - NOTE: Check each newlot of PVC filters by analyzing one or more by this method. For example, Gelman VM-1 filters (all lots) were found to be unacceptable because of high ash and background. If THF is used, check for complete dissolution by dissolving a blank PVC filter and following through steps 5c through 8.
 - b. Cyclone: 10-mm nylon, Higgins-Dewell (HD), Aluminum (Al), or equivalent.
- Area air sampler: PVC membrane filter, 37mm diameter, 5-μm pore size; three-piece filter cassette.
- Sampling pumps with flexible connecting tubing, capable of the following flow rates: nylon cyclone, 1.7 L/min; HD cyclone, 2.2 L/min; Al cyclone, 2.5 L/min; and bulk sampler, 3 L/min.
- 4. Silver membrane filters, 25-mm diameter, 0.45-µm pore size.
- 5. X-ray powder diffractometer (XRD) equipped with copper target X-ray tube, graphite monochromator, and scintillation detector.
- Reference specimen (mica, Arkansas stone, or other stable standard) for data normalization.
- 7. Low-temperature radio-frequency plasma asher (LTA) or muffle furnace, or ultrasonic bath (≥150 W), for filter preparation.
- Vacuum filtration assembly and side-arm vacuum flask with a 25-mm filter holder.
- 9. Sieve, 10-µm. for wet sieving.
- 10. Analytical balance (0.001 mg); magnetic stirrer with thermally insulated top; ultrasonic bath or probe; volumetric pipettes and flasks; Pyrex crucibles with covers (muffle furnace); 40-mL wide-mouth or 50-mL centrifuge tubes (THF method); desiccator; reagent bottles with ground glass stoppers; drying oven; polyethylene wash bottle.

SPECIAL PRECAUTIONS: Avoid inhaling silica dust [3]. THF isextremely flammable and should be used in a fume hood.

SAMPLING:

- 1. Calibrate each personal sampling pump with a representative sampler in line.
- 2. Sample at 1.7 \pm 5% L/min with nylon cyclone, 2.2 \pm 5% L/min with HD cyclone, ro 2.5 \pm 5% Al cyclone for a total sample size of 400 to 1000 L. Do not exceed 2 mg dust loading on the filter.
 - NOTE: Do not allow the sampler assembly to be inverted at any time when using a cyclone. Turning the cyclone to anythingmore than a horizontal orientation may deposit oversized material from the cyclone body onto the filter.
- 3. Take an area air sample or collect a settled dust sample, if dust in the work environment has not been previously characterized.

SAMPLE PREPARATION:

- 4. Samples may be characterized by one of the following methods, as appropriate.
 - a. **Interference check.** Prepare area dust sample or settled dust bulk sample for XRD analysis by mounting the collection sample directly on an XRD sample holder, or by depositing or redepositing the dust on another filter for mounting, or by packing an XRD powder holder. Proceed to step 11.
 - b. Qualitative Analysis. Prepare the area air sample or settled dust sample for qualitative analysis by grinding and/or wet sieving to best match the airborne dust particle size. Wet sieve with a 10-µm sieve, 2-propanol, and an ultrasonic bath [11], followed by evaporation of excess alcohol, drying in an oven for 2 hours, and overnight storage in a desiccator. Deposit the end product on a filter (steps 7-8) or pack in a conventional XRD powder holder.
 - NOTE 1: For quantitative determination of % SiQ, weigh out, in triplicate, 2 mg sieved dust, transfer to a 50-mL beaker, add 10 mL 2-propanol, and continue with step 6.
 - NOTE 2: In a bulk sample, if there is an interfering compound(s) that renders the identification and quantitation of quartz very difficult, the sample will need to be carefully treated in hot phosphoric acid [12] to dissolve the interfering compound(s) and avoid the loss of quartz. This treatment can be used to dissolve several 50-mg sample aliquots in order to concentrate the quartz content for the purpose of lowering the LOD.
- 5. Use one of the following methods to prepare filter samples and blanks:
 - a. Low Temperature Ashing: Place the filters in 50-mL beakers within the low temperature asher so that the sample exposure to the plasma is optimized. Ash according to manufacturer's instructions. After ashing, carefully add 15 mL 2-propanol to each beaker; or
 - b. Muffle Furnace Ashing:
 - 1. If the samples contain a significant amount of calcite (>20% of total dust loading), silica may be lost due to formation of CaSiQ. Remove the calcite by the following procedure: Place a 0.5-µm, 25-mm PVC filter in the filtration apparatus and clamp the filter funnel over it. Remove the sample filter from the cassette, fold, and drop it on the 25-mm filter. Add 10 mL 25% v/v HCl and 5 mL 2-propanol to the filter funnel and allow to sit for 5 min. Apply vacuum and slowly aspirate the acid and alcohol in the funnel, washing with three successive 10-mL portions of distilled water. Release the vacuum. Carry both filters through the ashing step together.
 - 2. Place the filter samples in porcelain crucibles, loosely cover and ash imuffle furnace for 2 h at 600 °C (800 °C if graphite is present). Add several mL 2-propanol to the ash, scrape the crucible with a glass rod to loosen all particles and transfer the residue to a 50-mL beaker. Wash the crucible several more times and add wash to beaker. Add 2-propanol to the beaker to bring the volume to about 15 mL; or
 - c. Filter Dissolution: Using forceps and a spatula, remove the filter from the cassette, fold the filter three times, and place in the bottom of a 40- or 50-mL centrifuge tube. Add 10 mL THF and allow to stand for at least 5 min. Cap the centrifuge tube with aluminum foil to prevent contamination. Gently agitate the centrifuge tube by hand or with avortex mixer making sure the THF does not go near the top of the tube. Place the tube in an ultrasonic bath (water level 2.5 cm from top) for at least 10 min. (The filter should be totally dissolved.) Just prior to filtering, agitate the sample for 10 to 20 sec on a vortex mixer. Continue with step 6, substituting THF for 2-propanol and centrifuge tube for beaker.
- 6. Cover the beaker with a watchglass and agitate in an ultrasonic bath for at least 3 min. Observe the suspension to make sure that the agglomerated particles are broken up. Wash the underside of the

- watchglass with 2-propanol, collecting the washings in the beaker.
- 7. Place a silver filter in the filtration apparatus. Attach the funnel securely over the entire filter circumference. With no vacuum, pour 2 to 3 mL 2-propanol onto the filter. Pour the sample suspension from the beaker into the funnel and apply vacuum. During filtration, rinse the beaker several times and add rinsings to the funnel.
- 8. Control the filtration rate to keep the liquid level in the funnel near the top during rinsing. Do not wash the walls or add 2-propanol to the funnel when the liquid level is lower than 4 cm above the filter. Leave the vacuum on after filtration to produce a dry filter. Remove the filter with forceps and mount it in the XRD sample holder.

CALIBRATION AND QUALITY CONTROL:

- 9. Prepare and analyze standard filters.
 - a. Prepare two suspensions of each analyte in 2-propanol by weighing 10 and 50 mg of the standard material to the nearest 0.01 mg. Quantitatively transfer each to a 1-L glass-stoppered bottle using 1.00 L of 2-propanol.
 - b. Suspend the powder in 2-propanol with an ultrasonic probe or bath for 20 min. Immediately move the flask to a magnetic stirrer with thermally insulated top and add a stirring bar. Cool to room temperature before withdrawing aliquots.
 - c. Mount a silver filter on the filtration apparatus. Place several mL of 2-propanol on the filter. Turn off the stirrer and shake vigorously by hand. Immediately remove the lid and withdraw an aliquot from the center at half-height of the 10 mg/L or 50 mg/L suspension. Do not adjust the volume in the pipet by expelling part of the suspension. If more than the desired aliquot is withdrawn, discard the aliquot in a beaker, rinse and dry the pipet, and take a new aliquot. Transfer the aliquot from the pipet to the silver filter, keeping the tip of the pipet near the surface but not submerged in the delivered suspension.
 - d. Rinse the pipet with several mL 2-propanol, draining the rinse into the funnel. Repeat the rinse several times.
 - e. Apply vacuum and rapidly filter the suspension. Do not wash down the sides of the funnel after the deposit is in place since this will rearrange the material on the silver filter. Leave vacuum on until filter is dry. Transfer the silver filter to the diffractometer sample mount. Prepare working standard filters, in triplicate, at e.g., 20, 30, 50, 100, 200, and 500 µg.
 - f. Analyze the working standards together with samples and blanks (step 12). The XRD intensities for the working standards (step 12.d) are designated are then normalized (step 12.e) to obtain Î_x. Correct the intensities of working standards >200 μg for matrix absorption (steps 12.f and 13).
 - g. Prepare a calibration graph (Ĵ, vs μg of each standard).
 - NOTE: Poor repeatability (>10% above 0.04 mg silica) at any given level indicates that new standards should be made. The data should lie along a straight line. A weighted least squares ($1/\sigma^2$ weighting) is preferable.
 - h. Determine the slope, m, of the calibration graph in counts/ μg . The intercept, b, on the abscissa should be within \pm 5 μg of zero.
 - NOTE: A large intercept indicates an error in determining the background, i.e., an incorrect baseline or interference by another phase.
- 10. NOTE: The following procedure for absorption is not necessary in situations that have been previously documented as requiring no corrections.
 - Select six silver membrane filters as media blanks randomly from the same box of filters to be used for depositing the samples. These will be used to test for sample self-absorption. Mount each of the media blanks on the filtration apparatus and apply vacuum to draw 5 to 10 mL 2-propanol through the filter. Remove, let dry, and mount on XRD holders. Determine the net normalized count for the silver peak, \hat{l}_{AG} , for each media blank (step 12). Obtain an average value for the six media blanks.

MEASUREMENT:

11. Obtain a qualitative X-ray diffraction scan (e.g., 10 to 802θ) of the area air sample (or bulk settled dust) to determine the presence of free silica polymorphs and interferences (see APPENDIX). The expected diffraction peaks are:

	Peak (2-Th		
Mineral	<u>Primary</u>	Secondary	<u>Tertiary</u>
Quartz	26.66	20.85	50.16
Cristobalite	21.93	36.11	31.46
Tridymite	21.62	20.50	23.28
Silver	38.12	44.28	77 47

NOTE: There is an alternative to scanning an area air sample, settled dust sample, or ground bulk sample to prove lack of contamination. A slow scan of the three main peaks of quartz (also cristobalite and tridymite if their absence has not been previously confirmed) on a personal air sample, and verification that their intensity ratios are within 15% of pure quartz, is sufficient evidence that other materials are not interfering in the silica determination.

- 12. Perform the following for each sample, working standard, and blank filter:
 - a. Mount the reference specimen. Determine the net intensity, lof the reference specimen before and after each filter is scanned. Use a diffraction peak of high intensity that can be rapidly but reproducibly (\$ < 0.01) measured.
 - b. Mount the sample, working standard, oblank filter. Measure the diffraction peak area for each silica polymorph. Scan times must be long, e.g., 15 min (longer scan times will lower the limit of detection).
 - c. Measure the background on each side of the peak for one-half the time used for peak scanning. The sum of these two counts is the average background. Determine the position of the background for each sample.
 - d. Calculate the net intensity, **!**, (the difference between the peak integrated count and the total background count).
 - e. Calculate and record the normalized intensity, if for each peak:

$$\hat{l}_x = \frac{l_x}{l_r} \cdot N.$$

NOTE: Select a convenient normalization scale factor, N, which is approximately equivalent to the net count for the reference specimen peak, and use this value of N for all analyses. Normalizing to the reference specimen intensity compensates for long-term drift in X-ray tube intensity. If intensity measurements are stable, the reference specimen may be run less frequently and the net intensities should be normalized to the most recently-measured reference intensity.

- f. Determine the normalized count, \hat{k}_g , of an interference-free silver peak on the sample filter following the same procedure. Use a short scan time for the silver peak (e.g., 5% of scan time for analyte peaks) throughout the method.
- g. Field blanks may be analyzed by scanning the 2-theta range used for the analyte and silver peaks to verify that contamination of the filters has not occurred. The analyte peak should be absent. The normalized intensity of the silver peak should match that of the media blank. Each laboratory should determine the specifics of field blank use for its application. When contamination does occur, the reason should be investigated and appropriate action taken. In practice, contamination of field blanks is extremely rare and usually is not consistent across filters. The analysis of blanks may be abbreviated if experience indicates that contamination is not likely with current field and laboratory operations; however, occasional confirmation of non-contamination is prudent.

CALCULATIONS:

13. Calculate the concentration of crystalline silica, C (mg/ft), in the air volume sampled, V (L):

$$C = \frac{\hat{i}_x \cdot f(t) - b}{m \cdot V}, mg/m^3$$

 \hat{l}_x = normalized intensity for sample peak

= intercept of calibration graph (Î vs. W)

m = slope of calibration graph, counts/μg

 $f(t) = -R \ln T/(1 - T^R) = absorption correction factor (Table 1)$

 $R = \sin(\Theta_{AG})/\sin(\Theta_{x})$

T = î_{Ag}/(average k̂_g) = transmittance of sample = normalized silver peak intensity from sample

 \hat{i}_{Ag} = normalized silver peak intensity from sample average \hat{f}_{Ag} = normalized silver peak intensity from media blanks (average of six values)

EVALUATION OF METHOD:

This method is based on P&CAM 259 which was collaboratively tested [1]. The testing included a ruggedization step to test the effects of the use of muffle furnace or plasma asher (but not the use of THF), shipment of samples, ashing time, and ultrasonication time. None of these factors was found to have an effect. The method was shown to have no bias when referenced to the Talvitie spectrophotometric method [10] and when all standards and samples were Min-U-Sil 5. The relative standard deviations (Sfor intralaboratory, total measurement and overall (including sampling) variability are:

Measurement Precision, S:

Intralaboratory: 50 to 200 µg -- 0.08 [1]

20 μg -- 0.20 [5] 10 μg -- 0.28 [9]

Total (intra-and interlaboratory): 50 to 200 µg -- 0.17 [1]

Overall (Sampling & Measurement) Precision \$\hat{S}_{rt}:

Total (intra- and interlaboratory): 50 to 200 μg -- 0.29 [1]

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APPENDIX: INTERFERENCES

Interferences include barite, micas (muscovite, biotite), potash, feldspars (microcline, plagioclase), montmorillonite, sillimanite, zircon, graphite, iron carbide, clinoferrosillite, wollastonite, sanidine, leucite, orthoclase, and lead sulfide.

The patterns for three forms of aluminum phosphate [JCPDS 10-423, 11-500, 20-44] are practically identical to those of quartz, cristobalite and tridymite, respectively. The quartz secondary and cristobalite primary peaks are close; cristobalite secondary peak is overlapped by a quartz peak; tridymite if present in sufficient quantity, will interfere with all of the main (primary, secondary and tertiary) quartz and cristobalite peaks. Silver chloride, if present on the silver filter, interferes slightly with the primary quartz peak. Many of these interferences occur in the presence of quartz; however, in a study of samples collected in 11 different industries, Altree-William [17] found no significant interferences.

The presence of elements such as iron can result in appreciable X-ray fluorescence which leads to high background intensity. A diffracted-beam monochromator will minimize this problem.

If calcite is present, loss of quartz will occur when samples are ashed in a muffle furnace. See SAMPLE PREPARATION (step 5.b) for procedure to remove calcite.

If interferences with the primary silica peak are present, use a less sensitive peak. When overlaps are not severe, a smaller receiving slitor chromium radiation may be used; however, a new calibration curve will be necessary.

Table 1. Absorption correction factor as a function of transmittance for some silica-silver peak combinations [13-18].

	f(T) (at indicated degrees 2-θ)									
Transmittance	Silica	26.66	26.66	20.83	20.83	21.93	21.93	21.62	21.62	
Т	Silver	38.12	44.28	38.12	44.28	38.12	44.28	38.12	44.28	•
1.00		1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	
0.99		1.0071	1.0082	1.0091	1.0105	1.0087	1.0100	1.0088	1.0101	
0.98		1.0144	1.0166	1.0184	1.0212	1.0174	1.0201	1.0177	1.0204	
0.97		1.0217	1.0251	1.0278	1.0321	1.0264	1.0305	1.0268	1.0309	
0.96		1.0292	1.0337	1.0373	1.0432	1.0355	1.0410	1.0360	1.0416	
0.95		1.0368	1.0425	1.0470	1.0544	1.0447	1.0517	1.0453	1.0524	
0.94		1.0445	1.0514	1.0569	1.0659	1.0541	1.0625	1.0548	1.0635	
0.93		1.0523	1.0605	1.0670	1.0776	1.0636	1.0736	1.0645	1.0747	
0.92		1.0602	1.0697	1.0772	1.0894	1.0733	1.0849	1.0743	1.0861	
0.91		1.0683	1.0791	1.0876	1.1015	1.0831	1.0963	1.0844	1.0977	
0.90		1.0765	1.0886	1.0982	1.1138	1.0932	1.1080	1.0945	1.1096	
0.89		1.0848	1.0983	1.1089	1.1264	1.1034	1.1199	1.1049	1.1216	
0.88		1.0933	1.1081	1.1199	1.1392	1.1137	1.1320	1.1154	1.1339	
0.87		1.1019	1.1181	1.1311	1.1522	1.1243	1.1443	1.1261	1.1464	
0.86		1,1106	1.1283	1.1424	1.1654	1.1350	1.1568	1.1370	1.1592	
0.85		1.1195	1.1387	1.1540	1.1790	1.1460	1.1696	1.1481	1.1722	
0.84		1.1286	1.1493	1.1657	1.1927	1.1571	1.1827	1.1595	1.1854	
0.83		1.1378	1.1600	1.1777	1.2068	1.1685	1.1959	1,1710	1.1989	
0.82		1.1471	1.1709	1.1899	1.2211	1.1800	1.2095	1.1827	1.2126	
0.81		1.1566	1.1821	1.2024	1.2357	1.1918	1.2232	1.1946	1.2266	
0.80		1.1663	1.1934	1.2150	1.2506	1.2038	1.2373	1.2068	1.2409	
0.79		1.1762	1.2050	1.2280	1.2658	1.2160	1.2516	1.2192	1.2555	
0.78		1.1863	1.2168	1.2411	1.2812	1.2284	1.2663	1.2319	1.2703	
0.77		1.1965	1.2288	1.2546	1.2971	1.2411	1.2812	1.2447	1.2855	
0.76		1.2069	1.2410	1.2683	1.3132	1.2540	1.2964	1.2579	1.3009	
0.75		1.2175	1.2535	1.2822	1.3297	1.2672	1.3119	1.2713	1.3167	
0.74		1.2283	1.2662	1.2965	1.3456	1.2806	1.3278	1.2849	1.3328	
0.73		1.2394	1.2792	1.3110	1.3637	1.2944	1.3440	1.2989	1.3493	
0.72		1.2506	1.2924	1.3259	1.3812	1.3084	1.3605	1.3131	1.3661	
0.71		1.2621	1.3059	1.3410	1.3991	1.3226	1.3774	1.3276	1.3883	
0.70		1.2738	1.3197	1.3565	1.4174	1.3372	1.3946	1.3424	1.4008	
0.69		1.2857	1.3337	1.3723	1.4362	1.3521	1.4122	1.3576	1.4187	
0.68		1.2979	1.3481	1.3885	1.4553	1.3673	1.4303	1.3730	1.4370	
0.67		1.3103	1.3682	1.4050	1.4749	1.3829	1.4487	1.3888	1.4558	
0.66		1.3230	1.3777	1.4218	1.4949	1.3987	1.4675	1.4050	1.4749	
0.65		1.3359	1.3931	1.4390	1.5154	1.4150	1.4868	1.4215	1.4945	
0.64		1.3491	1.4087	1.4567	1.5363	1.4316	1.5064	1.4383	1.5145	
0.63		1.3626	1.4247	1.4747	1.5578	1.4485	1.5266	1.4556	1.5350	
0.62		1.3765	1.4411	1,4931	1.5797	1.4659	1.5472	1.4732	1.5560	
0.61		1.3906	1.4578	1.5120	1.6022	1.4836	1.5684	1.4913	1.5775	
0.60		1.4050	1.4749	1.5314	1.6252	1.5018	1.5900	1.5098	1.5995	
0.59		1.4198	1.4925	1.5511	1.6488	1.5204	1.6122	1.5287	1.6221	
0.58		1.4349	1.5104	1.5714	1.6730	1.5394	1.6349	1.5481	1.6452	
0.57		1.4504	1.5288	1.5922	1.6978	1.5590	1.6582	1.5679	1.6689	
0.56		1.4662	1.5476	1.6135	1.7233	1.5790	1.6820	1.5883	1.6932	
0.55		1.4824	1.5670	1.6353	1.7494	1.5995	1.7065	1.6092	1.7181	
0.54		1.4991	1.6858	1.6577	1.7762	1.6205	1.7317	1.6306	1.7437	
0.53		1.5161	1.6071	1.6807	1.8037	1.6421	1.7575	1.6525	1.7699	
0.52		1.5336	1.6279	1.7043	1.8319	1.6642	1.7840	1.6751	1.7969	
0.51		1.5515	1.6493	1.7285	1.8609	1.6870	1.8112	1.6982	1.8246	
0.50		1.5699	1.6713	1.7534	1.8908	1.7103	1.8391	1.7220	1.8531	